

**QUALITY ASSURANCE PROJECT PLAN  
ADDENDUM**

**ADDITIONAL AND UNCHARACTERIZED SITES OPERABLE UNIT  
CRAB ORCHARD NWR  
MARION, ILLINOIS - WILLIAMSON COUNTY**

September 18, 2006

*Prepared by:*

ENTRIX, Inc.  
800 Hart Road, Suite 250  
Barrington, IL 60010

*Prepared on Behalf of:*

**General Dynamics Ordnance and Tactical System, Inc.**

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QUALITY ASSURANCE PROJECT PLAN  
ADDENDUM  
ADDRESSING  
CRUISE OPERATIONS

QUALITY ASSURANCE PROJECT PLAN  
ADDENDUM  
ADDITIONAL AND UNCHARACTERIZED OTHER CORROSION-INDIC  
CRUISE OPERATIONS  
MARION, ILLINOIS WILSON COUNTY

Prepared by: NorthStar Computer L.L.C., NTRC, Inc., & Consulting Engineers & Architects

Prepared for: U.S. EPA, Region 5, Regional Division - Chicago, IL

Approved By: <u>[Signature]</u>	<u>11/3/06</u>
Project Manager	Date
Approved By: <u>[Signature]</u>	<u>11/13/06</u>
NorthStar Computer L.L.C. QA Manager	Date
Approved By: <u>[Signature]</u>	<u>11/08/06</u>
U.S. EPA, Region 5 Project Manager	Date
Approved By: <u>[Signature]</u>	<u>11/08/06</u>
U.S. EPA, Region 5 QA Manager	Date
Approved By: <u>[Signature]</u>	<u>11/10/06</u>
Consulting Engineers & Architects Project Manager	Date
Approved By: <u>[Signature]</u>	<u>11/14/06</u>
NorthStar QA Project Coordinator	Date
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U.S. EPA Region 5 Project Coordinator	Date
Approved By: _____	_____
U.S. EPA Region 5 Quality Assurance Manager	Date
Approved By: <u>[Signature]</u>	<u>11/16/06</u>
U.S. EPA, Region 5 Quality Assurance Manager	Date
Approved By: <u>[Signature]</u>	<u>11/14/06</u>
U.S. EPA, Region 5 Quality Assurance Manager	Date

Approved by Nancy J. [Signature] 11/28/06  
U.S. EPA.  
REMEDIAL PROJECT MANAGER

**ERRATA PAGES TO QUALITY ASSURANCE PROJECT PLAN ADDENDUM  
ADDITIONAL AND UNCHARACTERIZED SITES OPERABLE UNIT  
CRAB ORCHARD NWR**



STATE OF WASHINGTON  
DEPARTMENT OF ECOLOGY

PO Box 488 • Manchester, WA 98353-0488 • (360) 895-6144

November 8, 2006

Mr. Dave Mitchell  
Analytical Resources, Incorporated  
4611 South 134th Place, Suite 100  
Tukwila, WA 98168-3240

Dear Mr. Mitchell:

Thank you for requesting accreditation for Organophosphorus Pesticides by 8270 SIM. Interim accreditation has been granted based on the submitted SOP and data package. Enclosed is your revised Scope of Accreditation.

Your Scope of Accreditation has also been updated based on recent PT results. Full accreditation has been restored for Cyanide, and accreditation has been made provisional for Color and PAHs.

**Please note that we have not received any PT results for Trihalomethanes by EPA 524.2 since March 2005. In order to continue accreditation for this parameter, you must submit acceptable results no later than December 29, 2006.**

If you have any questions concerning the accreditation of your lab, please contact Alan Rue at 360-895-6178, fax (360) 895-6180, or by e-mail at [arue461@ecy.wa.gov](mailto:arue461@ecy.wa.gov).

Sincerely,

Stewart M. Lombard  
Lab Accreditation Unit Supervisor

SL:AR:ar  
Enclosures



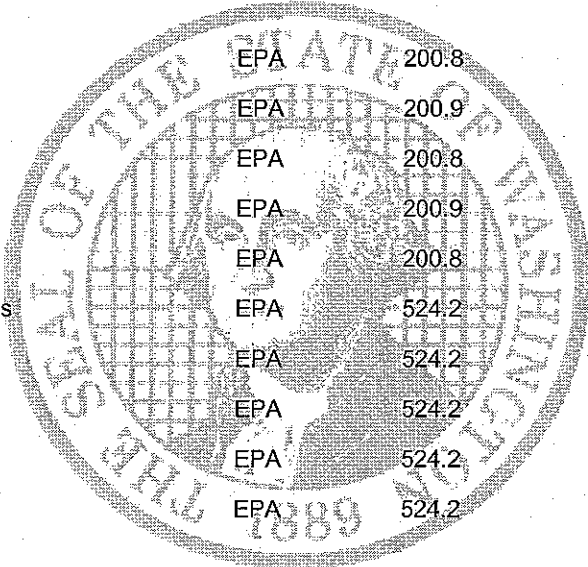
# Scope of Accreditation

## Analytical Resources, Incorporated

Tukwila, WA

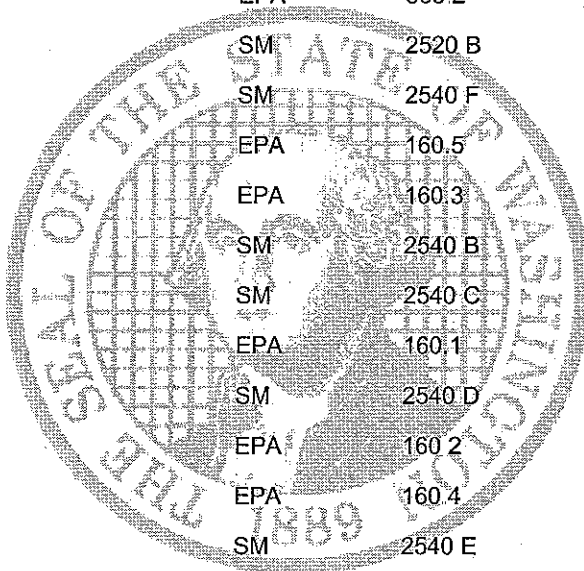
is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation may apply to any of the following matrix types: non-potable water, drinking water, solid and chemical materials, and air and emissions. Accreditation for all parameters is final unless indicated otherwise in a note. Accreditation is for the latest version of a method unless otherwise specified in a note. EPA refers to the U.S. Environmental Protection Agency. SM refers to American Public Health Association's publication, Standard Methods for the Examination of Water and Wastewater, 18th, 19th or 20th Edition, unless otherwise noted. ASTM stands for the American Society for Testing and Materials. PSEP stands for Puget Sound Estuary Program. Other references are detailed in the notes section.

Matrix Type/Parameter Name	Reference	Method Number	Notes
<b>Drinking Water</b>			
Arsenic	EPA	200.8	1
Arsenic	EPA	200.9	1
Copper	EPA	200.8	1
Lead	EPA	200.9	1
Lead	EPA	200.8	1
Purgeable Organic Compounds	EPA	524.2	1
Regulated VOCs	EPA	524.2	1
Trihalomethanes	EPA	524.2	1,8
Unregulated VOCs	EPA	524.2	1
Vinyl Chloride	EPA	524.2	1
<b>Non-potable Water</b>			
Acidity	EPA	305.1	1
Acidity	SM	2310 B(4c)	1
Alkalinity, Total	SM	2320 B(4c)	
Alkalinity, Total	EPA	310.1	
Ammonia	SM 19/20	4500-NH3 G	1
Ammonia	SM 19/20	4500-NH3 D	1
Ammonia	EPA	350.1	1
Ammonia	EPA	350.3	1

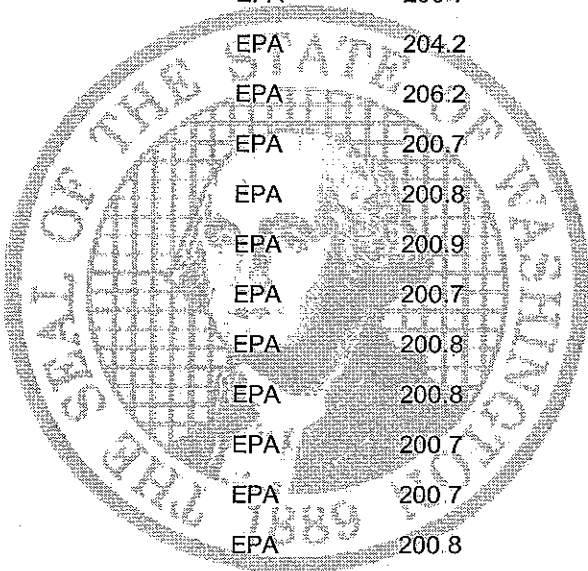


Matrix Type/Parameter Name	Reference	Method Number	Notes
Biochemical Oxygen Demand, BOD/CBOD	EPA	405.1	1
Biochemical Oxygen Demand, BOD/CBOD	SM	5210 B	1
Bromide	SM	4110 B	1
Bromide	EPA	300.0	1
Chemical Oxygen Demand (COD)	SM	5220 D	1
Chemical Oxygen Demand (COD)	EPA	410.4(7.3)	1
Chloride	SM	4110 B	1
Chloride	SM	4500-Cl-E	1,8
Chloride	EPA	325.2	1,8
Chloride	EPA	300.0	1
Chromium, Hexavalent	SM	3500-Cr D	1
Color	SM	2120 B	1,8
Color	EPA	110.2	1,8
Cyanide, Total	EPA	335.2(8.10)	1
Cyanide, Weak Acid Dissociable	SM	4500-CN I	
Cyanides, Amenable to Chlorination	SM	4500-CN G	1
Cyanides, Amenable to Chlorination	EPA	335.1	1
Dissolved Oxygen	SM	4500-O C	1
Dissolved Oxygen	EPA	360.2	1
Fluoride	EPA	300.0	1
Fluoride	EPA	340.2	1
Fluoride	SM	4110 B	1
Hardness, Total (as CaCO <sub>3</sub> )	SM	2340 B	1
Hexane Extractable Material	EPA	1664	1
Nitrate	EPA	353.2	1
Nitrate	SM	4500-NO <sub>3</sub> F	1
Nitrate	EPA	300.0	1
Nitrate	SM	4110 B	1
Nitrate + Nitrite	EPA	353.2	1
Nitrate + Nitrite	SM	4500-NO <sub>3</sub> F	1

Matrix Type/Parameter Name	Reference	Method Number	Notes
Nitrite	EPA	353.2	1
Nitrite	SM	4110 B	1
Nitrite	EPA	300.0	1
Nitrogen, Total Kjeldahl	EPA	351.4	1
Orthophosphate	EPA	365.2	
Orthophosphate	SM	4110 B	1
Orthophosphate	EPA	300.0	1
pH	EPA	150.1	1
Phenolics, Total Recoverable	EPA	420.1	1
Phosphorus, Total	SM	4500-P E	
Phosphorus, Total	EPA	365.2	1
Salinity	SM	2520 B	
Solids, Settleable	SM	2540 F	1
Solids, Settleable	EPA	160.5	1
Solids, Total	EPA	160.3	1
Solids, Total	SM	2540 B	1
Solids, Total Dissolved	SM	2540 C	1
Solids, Total Dissolved	EPA	160.1	1
Solids, Total Suspended	SM	2540 D	1
Solids, Total Suspended	EPA	160.2	1
Solids, Total Volatile	EPA	160.4	1
Solids, Total Volatile	SM	2540 E	
Specific Conductance	SM	2510 B	1
Specific Conductance	EPA	120.1	1
Sulfate	EPA	375.2	1
Sulfate	SM	4110 B	1
Sulfate	EPA	300.0	1
Sulfide	SM	4500-S2 D	1
Sulfide	EPA	376.2	1
Sulfide	SM	4500-S2 E	

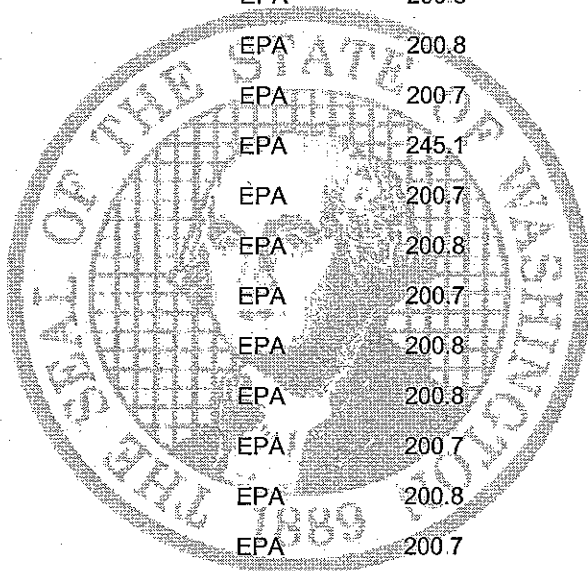


Matrix Type/Parameter Name	Reference	Method Number	Notes
Sulfide	EPA	376.1	
Sulfite	EPA	377.1	1
Total Organic Carbon	SM	5310 B	1
Total Organic Carbon	EPA	415.1	1
Turbidity	SM	2130 B	1
Turbidity	EPA	180.1	1
Aluminum	EPA	200.8	
Aluminum	EPA	200.7	1
Antimony	EPA	200.9	
Antimony	EPA	200.8	
Antimony	EPA	200.7	1
Antimony	EPA	204.2	1
Arsenic	EPA	206.2	1
Arsenic	EPA	200.7	1
Arsenic	EPA	200.8	
Arsenic	EPA	200.9	
Barium	EPA	200.7	1
Barium	EPA	200.8	
Beryllium	EPA	200.8	
Beryllium	EPA	200.7	1
Boron	EPA	200.7	1
Cadmium	EPA	200.8	
Cadmium	EPA	200.7	1
Cadmium	EPA	213.2	1
Cadmium	EPA	200.9	
Calcium	EPA	200.7	1
Calcium	EPA	200.8	
Chromium	EPA	200.8	
Chromium	EPA	200.7	1
Cobalt	EPA	200.8	



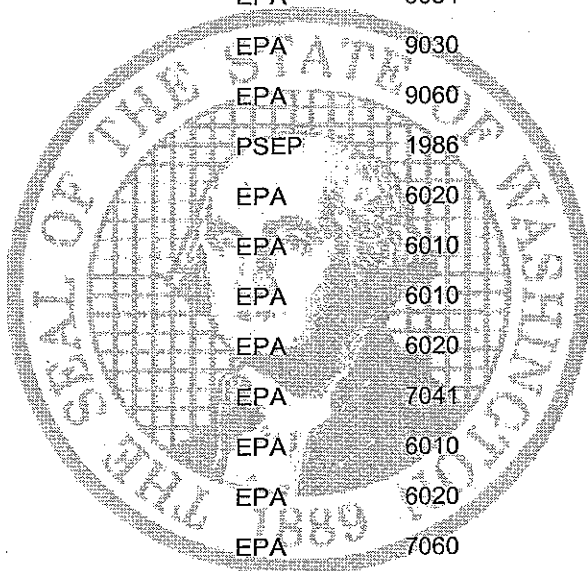


Matrix Type/Parameter Name	Reference	Method Number	Notes
Cobalt	EPA	200.7	1
Copper	EPA	200.7	1
Copper	EPA	200.8	
Iron	EPA	200.7	1
Iron	EPA	200.8	
Lead	EPA	200.8	
Lead	EPA	200.7	1
Lead	EPA	200.9	
Lead	EPA	239.2	1
Magnesium	EPA	200.7	1
Magnesium	EPA	200.8	
Manganese	EPA	200.8	
Manganese	EPA	200.7	1
Mercury	EPA	245.1	1
Molybdenum	EPA	200.7	1
Molybdenum	EPA	200.8	
Nickel	EPA	200.7	1
Nickel	EPA	200.8	
Potassium	EPA	200.8	
Potassium	EPA	200.7	1
Selenium	EPA	200.8	
Selenium	EPA	200.7	1
Selenium	EPA	200.9	
Selenium	EPA	270.2	1
Silica	EPA	200.7	1
Silica	SM	3120 B	
Silver	EPA	272.2	1
Silver	EPA	200.7	1
Silver	EPA	200.8	
Silver	EPA	200.9	

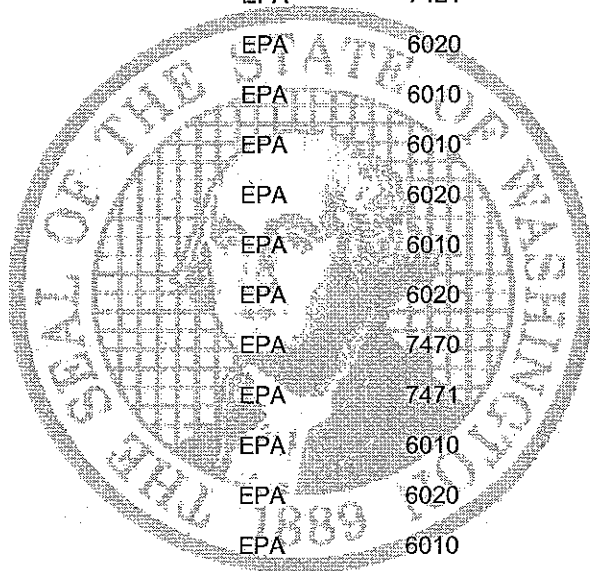


Matrix Type/Parameter Name	Reference	Method Number	Notes
Sodium	EPA	200.8	
Sodium	EPA	200.7	1
Strontium	EPA	200.7	1
Thallium	EPA	200.7	1
Thallium	EPA	200.8	
Thallium	EPA	200.9	
Thallium	EPA	279.2	1
Tin	EPA	200.7	1
Titanium	EPA	200.7	1
Uranium	EPA	200.8	
Vanadium	EPA	200.7	1
Vanadium	EPA	200.8	
Zinc	EPA	200.8	
Zinc	EPA	200.7	1
Methane, Ethane & Ethene	EPA	RSK-175	9,10
VOA & Semi-VOA Compounds	ARI SOP	427S	4
Butyl-tin species	ARI	802S	5
Vinyl Chloride/1,1-DCE	ARI	703S	2,6
Fecal Coliform - count	SM	9222 D	
Total Coli - count MF	SM	9222 B2,5,6	
<b>Solid and Chemical Materials</b>			
Bromide	EPA	9056	1
Chloride	EPA	9056	1
Chloride	EPA	9251	1
Chromium, Hexavalent	EPA	7196	1
Cyanide, Free	EPA	9014(7.2)	1
Cyanide, Total	EPA	9010	1
Cyanide, Total	EPA	9014(7.2)	1
Cyanides, Amenable to Chlorination	EPA	9010	1
Fluoride	EPA	9056	1

Matrix Type/Parameter Name	Reference	Method Number	Notes
Fluoride	EPA	9214	1
Hexane Extractable Material	EPA	9071 B	1
Nitrate	EPA	9056	1
Nitrite	EPA	9056	1
Orthophosphate	EPA	9056	1
pH	EPA	9040	
pH (non-aqueous)	EPA	9045	1
Phenolics, Total Recoverable	EPA	9065	1
Sulfate	EPA	9056	1
Sulfate	EPA	9036	
Sulfide	EPA	9034	
Sulfide	EPA	9030	1
Total Organic Carbon	EPA	9060	
Total Organic Carbon (Sed)	PSEP	1986	8
Aluminum	EPA	6020	
Aluminum	EPA	6010	1
Antimony	EPA	6010	1
Antimony	EPA	6020	
Antimony	EPA	7041	1
Arsenic	EPA	6010	1
Arsenic	EPA	6020	
Arsenic	EPA	7060	1
Barium	EPA	6010	1
Barium	EPA	6020	
Beryllium	EPA	6010	1
Beryllium	EPA	6020	
Boron	EPA	6010	1
Cadmium	EPA	6010	1
Cadmium	EPA	6020	
Cadmium	EPA	7131	1



Matrix Type/Parameter Name	Reference	Method Number	Notes
Calcium	EPA	6020	
Calcium	EPA	6010	1
Chromium	EPA	6010	1
Chromium	EPA	6020	
Cobalt	EPA	6010	1
Cobalt	EPA	6020	
Copper	EPA	6010	1
Copper	EPA	6020	
Iron	EPA	6020	
Iron	EPA	6010	1
Lead	EPA	7421	1
Lead	EPA	6020	
Lead	EPA	6010	1
Magnesium	EPA	6010	1
Magnesium	EPA	6020	
Manganese	EPA	6010	1
Manganese	EPA	6020	
Mercury, Liquid Waste	EPA	7470	1
Mercury, Solid Waste	EPA	7471	1
Molybdenum	EPA	6010	1
Molybdenum	EPA	6020	
Nickel	EPA	6010	1
Nickel	EPA	6020	
Potassium	EPA	6010	1
Potassium	EPA	6020	
Selenium	EPA	6020	
Selenium	EPA	7740	1
Selenium	EPA	6010	1
Silica	EPA	6020	
Silica	EPA	6010	1

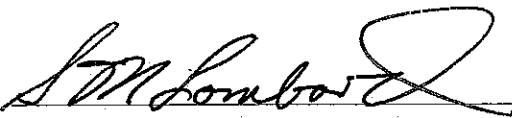


Matrix Type/Parameter Name	Reference	Method Number	Notes
Silver	EPA	6010	1
Silver	EPA	6020	
Silver	EPA	7761	1
Sodium	EPA	6020	
Sodium	EPA	6010	1
Strontium	EPA	6010	1
Thallium	EPA	6020	
Thallium	EPA	7841	1
Thallium	EPA	6010	1
Tin	EPA	6010	1
Titanium	EPA	6010	1
Uranium	EPA	6020 Mod	
Vanadium	EPA	6020	
Vanadium	EPA	6010	1
Zinc	EPA	6020	
Zinc	EPA	6010	1
Chlorinated Herbicides	EPA	8151	1
Organochlorine Pesticides	EPA	8081	1
Petroleum Hydrocarbons, Extractable	WDOE	EPH	1
Petroleum Hydrocarbons, Volatile	WDOE	VPH	1
Phenols	EPA	8041	
Polychlorinated Biphenyls	EPA	8082	1,7
Total Pet Hydrocarbons - Diesel	WDOE	NWTPH-Dx	1,3
Total Pet Hydrocarbons - Gasoline	WDOE	NWTPH-Gx	1,3
Volatile Aromatics	EPA	8021	1
BNA Extr (Semivolatile) Organics	EPA	8270	1
Organophosphorus Pesticides	EPA	8270	2,10
Polycyclic Aromatic HC (PAHs)	EPA	8270 SIM	1,2,8
Volatile Organic Compounds	EPA	8260	1
Particle Size Distribution	ASTM	D 422	

Matrix Type/Parameter Name	Reference	Method Number	Notes
Particle Size Distribution (Sed)	PSEP	1986	

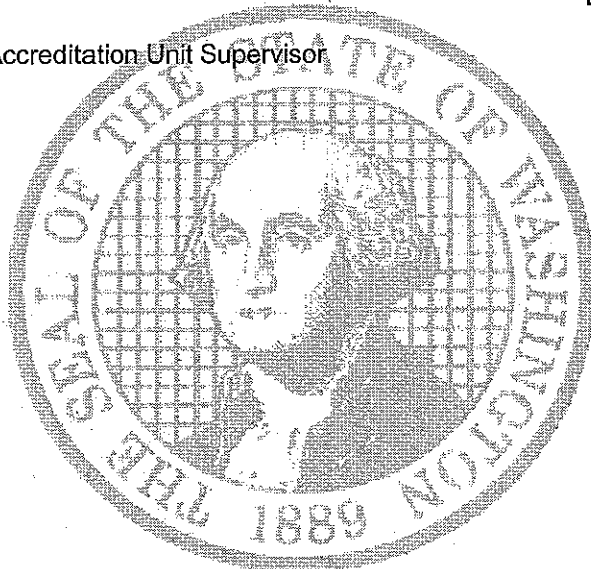
**Accredited Parameter Note Detail**

(1) Recognition of Oregon NELAP accreditation. (2) GC-MS Selective Ion Monitoring (SIM). (3) Washington Department of Ecology Analytical Methods for Petroleum Hydrocarbons, Publication Number ECY 97-602, June 1997. (4) ARI SOP for "Water Soluble Non-halogenated Volatile and Semivolatile Organic Compounds," including glycols. (5) ARI SOP, a modification of Krone Procedure. (6) ARI SOP for Vinyl Chloride determination. (7) Includes capability for low levels in aqueous samples using a modified hexane extraction. (8) Provisional pending an acceptable proficiency testing (PT) result (WAC 173-50-110). (9) ARI SOP 710S. (10) Interim pending on-site assessment to verify analytical capability.

  
 Authentication Signature

*November 8, 2006*  
 Date

Stewart M. Lombard, Lab Accreditation Unit Supervisor



## Parameters Denied Accreditation

### Analytical Resources, Incorporated

Tukwila, WA

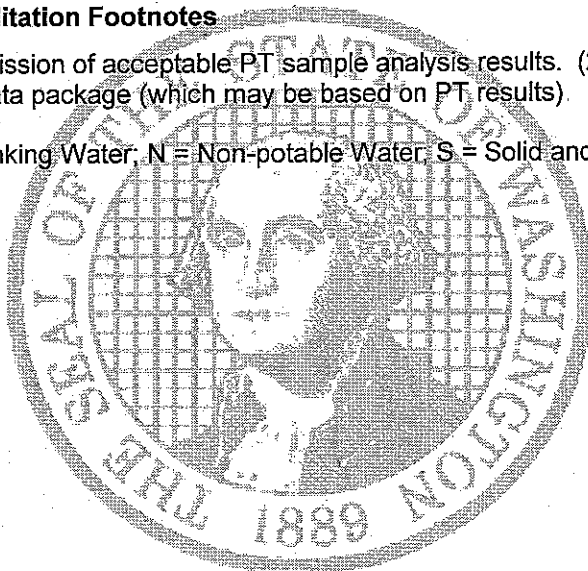
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Parameter Name	Reference	Method Number	Notes	Matrix
Enterococcus/Fecal Strep	SM	9230 C2ab,3,4,5	2	N
Heterotrophic Plate Count	SM	9215 D	2	N
Strontium	EPA	200.8	1	N
Strontium	EPA	6020	1	S
Tin	EPA	200.8	1	N
Titanium	EPA	200.8	1	N
Titanium	EPA	6020	1	S

#### Denied Parameter Accreditation Footnotes

(1) Withheld pending submission of acceptable PT sample analysis results. (2) Withheld pending acceptable lab SOP and data package (which may be based on PT results).


Matrix Definitions - D = Drinking Water; N = Non-potable Water; S = Solid and Chemical Material; A = Air and Emissions.



**Washington State Department of Ecology  
Environmental Assessment Program  
Lab Accreditation Unit**

Technical Report

For: Analytical Resources, Inc., Tukwila, Washington

Author: Alan Rue, Lab Accreditation Unit 

Date: November 3, 2006

Re: SOP for Organophosphorus Pesticides by GC/MS SIM

The section numbers below refer to ARI SOP 805S, Revision 001, 8/25/06, which should be revised as indicated:

3.1, 3.3, 3.6, 3.7, 3.13, 3.15, 3.17 Terms are missing definitions.

6.8 Derivatization apparatus and reagents – used for this analysis? If not, delete from SOP.



November 20, 2006

Ms. Leanne Moore  
U.S. Fish and Wildlife Service  
8588 Route 148  
Marion, IL 62959

**RE: Response to Informal Agency Comments to the Revised Addendum – AUS OU, Crab Orchard National Wildlife Refuge NPL Site.**

Dear Ms. Moore:

Enclosed you will find a response revision to address comment #3 of the Informal Agency Comments to the Revised QAPP Addendum – AUS OU.

*Comment 3: It is unclear what the acceptance criteria will be for the Organic free water identified in Section 9.0. Please verify the QC objectives are sufficient to meet the project objectives. It is important the QAPP clearly identify the specific criteria.*

Since it is recognized that there is a need to have a structured process for dealing with the Organic Free Water and its ensured purity, the following quality assurance steps have been proposed:

- **All Organic free water will be contained in clean glass, which has been dedicated for organic free water storage.**
- **If the organic free water is being obtained from a purification system, at the initial set up, a sample will be taken that will provide the water background and makeup. From this point on one sample per week will be taken and sent to STL to be analyzed for the project specific volatile and metal compounds. The initial sample will be used as the background comparison for the future samples. A sample will also be taken at the changing of the cartridge.**
- **Water that is supplied by the laboratory is certified as pure when received from Fischer. The laboratory will provide certification for each lot of water upon request.**
- **Equipment blanks will also be utilized to monitor for any potential problems with the organic free water quality. If the Equipment Blank shows signs of contamination, the weekly organic free water analytical results will be reviewed. In the case of the laboratory supplied water the purity certification will be reviewed.**
- **If it is found that the likely source is the purification system, then use of the purification system water will be halted and organic free water from the lab will be used until the problem is rectified.**
- **If the laboratory is providing the organic free water being used, then an alternative source will be used until the problem is rectified.**

Having these processes in place will enable us to monitor the water and correct any issues that may arise quickly and proactively. It is important to also note that to date 72,800 sample points have gone through validation by Ecochem (the third-party validator). Of those 72,800 sample points Ecochem has qualified approximately .03% (or a total of 19 sample points) due to equipment blanks. The process outlined above will help to ensure, to the extent possible, that this continues to hold true (as far as organic free water is concerned) throughout the life of the project.

Please except this as the revised response to comment #3. Should there be any questions, please feel free to contact Dave Trainor at the Madison, Wisconsin New Fields office (608) 442-5223 or myself (847) 277-2850. We look forward to your comments, and appreciate your time.

Respectfully,

Cheryl Randle  
Senior Quality Assurance Coordinator  
Entrix, Inc.



ENTRIX, Inc.  
800 Hart Road, Suite 250  
Barrington, IL 60010  
(847) 277-2850 | (Fax) (847) 381-6679  
[www.entrix.com](http://www.entrix.com)

November 30, 2006

Ms. Leanne Moore  
U.S. Fish and Wildlife Service  
8588 Route 148  
Marion, IL 62959

**RE: Response to Informal Agency Comment No. 5 – AUS OU, Crab Orchard National Wildlife Refuge NPL Site.**

Dear Ms. Moore:

Enclosed you will find an errata and revised response to comment #5 of the Informal Agency Comments to the Revised QAPP Addendum – AUS OU.

*Comment 5: TCL is referenced which typically refers to the CLP SOW Target Compound List (TCL), which does not include any of the organophosphorus pesticides (OPPs) or herbicides. Suggest removing TCL reference.*

Since TCL can commonly be in reference to the CLP SOW Target Compound List, and the methodology being utilized for the organophosphorous pesticides (OPPs) or herbicides are SW846 not CLP, any reference made to TCL has been removed from the QAPP Addendum document as to avoid further confusion.

Please except this errata and revised response to comment #5. Should there be any questions, please feel free to contact Dave Trainor at the Madison, Wisconsin New Fields office (608) 442-5223 or myself (847) 277-2850. We look forward to your comments, and appreciate your time.

Respectfully,

Cheryl Randle  
Senior Quality Assurance Coordinator  
Entrix, Inc.

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## LIST OF ACRONYMS/ABBREVIATIONS

QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation Feasibility Study
NWR	National Wildlife Refuge
ARI	Analytical Resources, Inc.
CSM	Client Services Manager
COC	Chain of Custody
QA	Quality Assurance
QAQC	Quality Assurance Quality Control
SOP	Standard Operating Procedure
RSD	Relative Standard Deviation
RPD	Relative Percent Difference
SMC	System Monitoring Compounds
LCS	Laboratory Control Sample
MS	Matrix Spike
MSD	Matrix Spike Duplicate
SRM	Standard Reference Materials
MDL	Method Detection Limit
USFWS	United States Fish and Wildlife Service
CLP	Contract Laboratory Program
DFTPP	Decafluorotriphenylphosphine
GCMS	Gas Chromatography Mass Spectrometer
USEPA	United States Environmental Protection Agency
NIST	National Institute of Standards
CCAL	Continuing Calibrations
CVS	Calibration Verification Standards
TCL	Target Compound List
GCPID	Gas Chromatography Flame Photometric Detector
SDG	Sample Delivery Group
EDD	Electronic Data Deliverable
AUS	Additional Uncharacterized Sites
FSP	Field Sampling Plan

## **LIST OF TABLES**

Table 1 – Ecological Benchmarks, Method Detection Limits, and Reporting Limits for Organophosphate Pesticides in an Aqueous Matrix

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## **LIST OF APPENDICES**

Appendix A – Laboratory SOPs

Appendix B – Laboratory Certifications

Appendix C – Laboratory QA Manual

## **1.0 QAPP ADDENDUM**

This Quality Assurance Project Plan (QAPP) addendum is intended to be used in conjunction with the EPA-approved *Quality Assurance Project Plan, Crab Orchard Additional and Uncharacterized Sites Operable Unit, Crab Orchard NWR, Marion Illinois, Williamson County, FINAL April 2006* (Entrix 2006) which was developed for the Crab Orchard National Wildlife Refuge Additional Uncharacterized Sites Remedial Investigation/Feasibility Study RI/FS. This QAPP addendum was prepared to specifically address the addition of Organophosphorous Pesticides and Herbicide compounds and to supply information on the laboratory that will support the analysis of these compounds. This QAPP addendum is also being utilized to address the following issues:

- Change in distribution address of Ecochem, Inc., the third party validator.
- The addition of a water purification system as the source of organic free water at the site.
- Adjustment made to Figure 9.1 of the QAPP; Data Validation Flowchart, Crab Orchard NWR
- Adjustments made to Figure 2.1 of the QAPP; Project Organizational Chart
- Additional information and clarification of the sample identification format found in Section 5.1.1 of the QAPP.

## **2.0 ADDITION OF A NEW LABORATORY**

### **2.1 Laboratory Responsibilities**

Analytical Resources, Inc. (ARI) has been selected as the analytical laboratory for the Organophosphorous Pesticides and Herbicide analysis portion of this project. Certificates for pertinent accreditation can be found in Appendix B. ARI is located at:

4611 South 134<sup>th</sup> Place  
Tukwila, Washington 98168-3240  
(206) 695-6200

The various QA and management responsibilities of key project personnel are defined below:

Analytical Resources, Inc. –Client Services Manager (CSM) – Susan D. Dunnihoo

The ARI CSM will communicate directly with the ENTRIX Project Manager and will be responsible for the following:

- Scheduling sample analyses with the laboratory;

- Verifying COC and accepted samples versus the Project QAPP;
- Relaying technical issues with the ENTRIX Project Manager;
- Overseeing data completeness;
- Overseeing preparation of final data package; and
- Approving final data package prior to distribution.

Analytical Resources, Inc., - QA Officer – David R. Mitchell

The ARI QA Officer will be responsible for the following:

- Overview laboratory QA;
- Overview QA/QC documentation;
- Overview in-house chain-of-custody (COC);
- Review laboratory corrective actions;
- Conduct detailed data review, if corrective action warrants;
- Review Laboratory Standard Operating Procedures (SOPs).

Analytical Resources, Inc. - Sample Custodian – Brian Kegel

Responsibilities of the ARI Sample Custodian include:

- Supervise the staff receiving and inspecting the incoming sample containers;
- Assure staff records the condition of the incoming sample containers;
- Assure signatures are obtained for appropriate documents;
- Assure staff completes the reverifying samples against COC;
- Notifying CS manager of sample receipt and inspection;
- Supervise staff assigning unique identification number and customer number to samples in the LIMS;
- Supervise sample transfer to secure storage, where analysts retrieve and document their removal for use in analysis. The sample custodian is also in charge of archiving the samples after they are returned by the analysts, and documents its arrival to keep a record of custody.
- Controlling and monitoring access/storage of samples.

Analytical Resources, Inc. – Organics Laboratory Manager – Brian Bebee

Responsibilities of the ARI Organics Laboratory Manager or his designee include:

- Manage the staff responsible for sample preparation and analysis;
- Assure staff completes re-verifying the incoming sample containers before preparation;
- Define appropriate laboratory QA procedures; and
- Prepare laboratory Standard Operating Procedures (SOPs) for the section;
- Oversee preparation of analytical reports; and
- Perform final data review of analyses.

#### Analytical Resources, Inc. Organics Technical Staff

The ARI Technical Staff will be responsible for sample preparation and analysis, scheduling sample analysis, identification of corrective actions and archival of extracts. The analyst is responsible for the initial data review, followed by a review from a peer. The Laboratory Manager reviews the data for final approval. The CSM/Project Manager reviews for completeness. The QA Officer will review any corrective action necessary.

### **3.0 Quality Assurance Objectives**

#### **3.1 Precision**

##### *3.1.1 Definition*

Precision is the degree of agreement among repeated measurements of the same characteristic (analyte, parameter, etc.) under the same or similar conditions (USEPA, 2000).

##### *3.1.2 Laboratory Precision Objectives*

Precision in the laboratory is assessed through RSDs (for three or more replicate samples) and the calculation of RPDs. The equations to be used for precision in this project can be found below. Precision control limits are included in the ARI SOPs found in Appendix A of this Addendum.

#### **3.2 Accuracy**

##### *3.2.1 Definition*

Accuracy is the extent of agreement between an observed value (sample results) and the accepted, or true, value of the parameter being measured (USEPA, 2000).



### 3.2.3 *Laboratory Accuracy Objectives*

Laboratory accuracy is assessed through the analysis of System Monitoring Compounds (SMC), Laboratory Control Samples (LCS), Matrix Spikes (MS), Matrix Spike Duplicates (MSD) or Standard Reference Materials (SRM) and the determination of percent recoveries (%R). The data generated demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The (%R) is calculated according to the following formula (USEPA, 2000):

$$\%R = \frac{\text{Spiked Sample Concentration} - \text{Unspiked Sample Concentration} \times 100}{\text{Concentration of Spike Added}}$$

SOPs for laboratory analyses are provided in Appendix A and contain the required accuracy, precision, sensitivity of the analyses.

## 3.3 **Completeness**

### 3.3.1 *Definition*

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

### 3.3.2 *Laboratory Completeness Objectives*

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. In this case completeness refers to all measurements that correspond to the Organophosphorous Pesticides and Herbicide analyses. The equation for completeness is presented below. Laboratory completeness for this project, and these analyses, should be 90 percent or greater.

$$\text{Completeness} = \frac{(\text{number of valid measurements}) \times 100}{(\text{number of measurements planned})}$$

## 3.4 **Representativeness**

### 3.4.1 *Definition*

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

### 3.4.2 *Measures to Ensure Representativeness of Laboratory Data*

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples.

## 3.5 **Comparability**

### 3.5.1 *Definition*

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

### 3.5.2 *Measures to Ensure Comparability of Laboratory Data*

The criteria for laboratory data comparability will be to ensure that the analytical methods used for the RI sampling and analysis events are comparable to the methods used for previous sampling events, if applicable.

## 3.6 **Sensitivity**

### 3.6.1 *Definition*

Sensitivity is the ability of the method or instrument to detect the contaminant of concern and other target compounds at the level of interest.

### 3.6.2 *Sensitivity of Laboratory Data*

ARI will evaluate and monitor method and instrument sensitivity through the development of laboratory method detection limits. A laboratory fortified blank, a blank that is spiked at the quantitation limit, is used in the development of the MDLs. Sensitivity is measured by calculating the percent recovery of the analytes at the quantitation limit. The SOP for MDL development has been provided in Appendix A of the QAPP and includes formulas for calculating analytical sensitivity. MDL studies are conducted on an annual basis, a standard in the laboratory industry. Since changes in limits can occur over the life of a project, any changes in the MDLs as a result of these studies, or for any other reason, will be reported to USFWS via the Quality Control Summary Report.

The level of QC effort provided by the laboratory will be equivalent to the level of QC effort specified under the contract laboratory program (CLP) for the Routine Analytical Service parameters to be tested. All data packages submitted to ENTRIX and NewFields will be Level IV, CLP equivalent. Please note that CLP is simply referenced as such. SW-846 methods will be used.

## **4.0 Calibration Procedure and Frequency**

### **4.1 Laboratory Instrument Calibration**

#### *4.1.1 OP-Pesticide Analysis by 8270D*

Prior to calibration, the instrument(s) used for Gas Chromatograph/Mass Spectrometer (GC/MS) are tuned by analysis of decafluorotriphenylphosphine (DFTPP). The instrument tune will be verified each 12 hours of operation. Once the tuning criteria are met, the instrument will be initially calibrated by using a minimum five-point calibration curve, and the calibration verified using a second source standard. The calibration standards will be USEPA or National Institute of Standards and Technology (NIST) traceable and are spiked with internal standards and surrogate compounds. Calibration standards used as reference standards will be traceable to the NIST or USEPA when existent.

Continuing calibrations (CCAL) are analyzed as specified in the method, or at least each working day, using criteria specified by the method.

#### *4.1.2 Herbicide Analysis by method 8151*

The instrument will be initially calibrated by using a minimum five-point calibration curve, and the calibration verified using a second source standard. The calibration standards will be USEPA or National Institute of Standards and Technology (NIST) traceable and are spiked with surrogate compounds.

Calibration Verification Standards (CVSs) must be run at the start and end of a run sequence and after every 10 sample injections within an analytical sequence and must meet the minimum requirements of the method. No more than 12 hours may elapse between the injection of the opening CVS and the closing CVS.

## **5.0 Analytical Methods**

### **5.1 Description - Organophosphorous Pesticides**

TCL organophosphorus pesticides will be extracted using separatory funnel extraction for aqueous samples and sonication for solid sample extraction. Aqueous samples are extracted at neutral pH with methylene chloride. Solid samples are extracted twice with methylene chloride/acetone (1:1) followed by one extraction with methylene chloride only. The extracts are analyzed using gas chromatography/mass spectrometer (GC/MS).

## **5.2 Description - Herbicides**

TCL Herbicides will be extracted using separatory funnel extraction for aqueous samples and sonication for solid samples. Aqueous samples are extracted using Hydrolysis followed by Separatory funnel extraction at a pH of <2.0 with Methylene Chloride/Acetone (70:30) followed by Methylene Chloride/Hexane (70:30). Solid samples are extracted with Methylene Chloride/acetone (70:30), followed by Hexane/Methylene Chloride (70:30) using Sonication, followed by Hydrolysis, with a clean-up and back-extraction. Extracts are analyzed using dual column GC/ECD.

## **5.3 Quantitation Limits and ESV'S**

Method detection limits, reporting limits, and ecological benchmarks can be found in this QAPP Addendum as tables 1, 2, 3 and 4.

## **6.0 Laboratory Data Reduction Procedures**

Laboratory data reduction procedures will be followed according to the following protocol. All references to raw analytical data will be recorded in numerically identified laboratory notebooks. These notebooks will be issued by the ARI QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP#), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the ARI QA Manager takes place prior to final data reporting. (Records of notebook entry inspections are maintained by the ARI QA Manager.)

QC data (e.g., laboratory duplicates, SMCs, SRM, MSs, and MSDs) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the ARI QA Manager for review. If approved, data are logged into the project database format. The laboratory shall appropriately flag unacceptable data in the data package.

At ARI, raw analytical data requiring manual documentation are recorded on benchsheets associated with each project file. Data are recorded on each benchsheet along with other pertinent information, such as the sample identification number and the sample tag number. Other details are recorded on each benchsheet, such as the analytical method used, name of analyst, the date of analysis, matrix sampled, reagent ID and concentrations,. Each benchsheet shall be signed and dated by the analyst and any spike witnesses. Bound notebooks are maintained for run logs for each instrument, and each page is signed by the analyst. Copies of all other raw data (tunes, calibrations,

quantitation reports, chromatograms, analyst notes, requests for re-extracts, LIMS worklists for data entry or data imports) will be maintained in each project file and electronically.

QC data (e.g., tunes, calibrations, laboratory duplicates, SMCs, SRM, MDs, MSs, and MSDs) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into LIMS worklists and peer reviewed. LIMS worklists along with the associated raw data will be submitted to the ARI Organic Manager or designee for review. If approved, data are distributed into the LIMS database for generation of final reports and EDDs. As referenced in section 9.1 of the QAPP, the Region 5 EDD format will be used for this project. An example of the EDD format can be found at the following web address: <http://www.epa.gov/superfund/programs/clp/sedd.htm>.

## 6.1 Chemistry Data Reporting

Case narratives will be prepared to include information concerning data that fell outside laboratory acceptance limits, and any other anomalous conditions encountered during sample analysis. The CLP equivalent Level IV data package shall include the following data elements:

### 1. Case Narrative:

- Any deviations from intended analytical strategy
- Laboratory lot number/sample delivery group (SDG)
- Numbers of samples and respective matrices
- QC procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples 'as-received'
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the QA Manager

### 2. Chemistry Data Package

- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory QC checks
- Cross referencing of laboratory sample to project sample identification numbers

- Data qualifiers to be used should be adequately described
- Sample preparation and analyses for samples
- Sample results
- Raw data for sample results and laboratory QC samples
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning results
- MS and MS duplicate recoveries, laboratory control samples, method blank results, calibration check compound, and system performance check compound results
- Labeled (and dated) chromatograms/spectra of sample results and laboratory QC checks
- Preparation factors and logbook notations

The laboratory shall also prepare and verify an EDD. The format of the EDD shall be in the approved Region 5 format.

1. For this investigation, ARI will provide a standard or 21-day turn-around-time for the analytical data package and EDD. The 21-day timeframe begins the day the ARI receives a given sample for analysis.

## **7.0 PREVENTIVE MAINTENANCE**

### **7.1 Preventative Maintenance Procedure**

Prior to the analysis of an initial calibration sequence, the following procedures should be followed.

- If needed, change the glass wool and, possibly, the injector liner itself.
- If needed, change the septum. Some GC-ECD instruments use “Merlin” mechanical septa to minimize retention time drift. These should be changed after 2000 injections (approximately), or sooner if drift is noticed and a Merlin leak is verified. Normal GC septa should be replaced at least once per 150 injections. Changing the septum during an analytical sequence should be avoided, if possible. If a septum is changed during a run, immediately run a standard to verify retention times and response factors.
- Check all column connections and injector/detector fittings.
- Refill solvent rinse vials.
- Bake the GC oven at 275° - 310°C (depending on the column(s) installed) for at least half an hour.
- Gas cylinder traps should be replaced annually.
- ECD detectors should be wipe tested every six months for radiation leaks

- Clean out cooling fans annually
- Check or replace diffusion pump fluid annually (GC/MS)
- Lubricate sideplate or vent valve O-rings annually (GC/MS)
- Calibration vial should be check annually (GC/MS)
- Foreline pump oil for should be checked annually (GC/MS)

## 8.0 SAMPLING PROCEDURES

The sampling procedures to be used in this investigation have been selected to achieve the goals of the DQOs outlined in Section 1 of the QAPP. The RI/FS Work Plan and FSP outline all the sampling procedure information.

### 8.1 Sample Identification

The sample identification format for this investigation has been adjusted for smoother use throughout the life of the project. The ID is designed to uniquely identify each sample from each sampling program and event. This portion of this QAPP Addendum has been written to satisfy the adjustments that have been made and replace that which has been detailed in Section 5.1.1 of the QAPP. An example of the sample format is as follows, and is described below:

**"AUS-OA2B-004-SL-05"**

- AUS-\_\_-\_\_-\_\_-\_\_: All samples will have the "AUS" prefix;
- \_\_-OA2B-\_\_-\_\_-\_\_: The second part of the number identifies the AUS OU site. The example describes Area 2B;
- \_\_-\_\_-004-\_\_-\_\_: The third part of the number identifies the sample location within that area;
- \_\_-\_\_-\_\_-SL-\_\_: The fourth part of the number identifies the media (i.e., SS=soil; SD=sediment; GW=groundwater; SW=surface water; SL=sludge). The fourth part also identifies a QC sample where appropriate (EB=equipment blank; TB=trip blank)
- \_\_-\_\_-\_\_-\_\_-0005: The last part of the number identifies the sample depth in feet, to the nearest foot of the bottom of the sample interval. For example if the sampling depth is 0-.5'; then the last four digits would read "0005". If the sampling depth is 6'-10'; then the last four digits would read "0610". If there is a case where the depth is not applicable (i.e. surface water) the last four digits would read "0000".
- For samples of waste material and samples collected from drums, the fourth and fifth positions will be replaced by a four-digit descriptor such as "drum" for a drum sample.

To ensure that duplicate samples cannot be distinguished from the original sample at the laboratory, a duplicate sample will hold all of the same information as its parent sample with the exclusion of the four-digit descriptor in the 5<sup>th</sup> position. Duplicate samples along with all other field QC sample locations will be numbered sequentially in the 5<sup>th</sup> position, starting with 3000 for sampling performed by Entrix, and 5000 for sampling performed by CRA. The notes taken in the field log will detail the original sample for which the duplicate has been taken.

## 8.2 Sample Containers, Sample Preservation and Maximum Holding Times

Samples collected for analysis will be contained and preserved in accordance with USEPA approved procedures.

All sample containers used for sample collection and analysis for this project will be prepared according to the procedures contained in the USEPA document, *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers*, dated December 1992. This document specifies the acceptable types of containers, the specific cleaning procedures to be used before samples are collected, and QA/QC requirements relevant to the containers and cleaning procedures. ARI and/or STL will supply all sample containers utilized for this investigation. If field personnel observe any cracked, dirty, or the appropriate preservative missing in the sample bottles, those bottles will be discarded and ARI and/or STL will be notified of the problem to prevent its re-occurrence.

The following provides a discussion of information provided in that table, presented by matrix and analytical groups.

### 8.2.1 Sample Containers for Solid Matrix

Solid matrix samples for this investigation include soils and sediments. These samples will be submitted to ARI for the following analyses: organophosphorous Pesticides and Herbicides.

- a. Soil samples for Organophosphorous Pesticides and Herbicides analysis will be collected in one 8oz glass jar for both analyses, and stored and transported to the laboratory at 4 degrees Celsius (C)  $\pm 2$  degrees C. Soil samples for these analyses have 14 days to extraction and 40 days thereafter for analysis.
- b. Sediment samples will be collected with a sediment corer whenever possible. If the depth of sediment is insufficient for discrete samples, composite samples will be collected as described in Section 4.1.1 of the FSP. After the sample is collected, it will be placed in a 8-ounce glass jar and preserved at 4 degrees C  $\pm 2$  degrees C, with a 14-day holding time before extraction and analysis.



### 8.2.2 *Sample Containers for Aqueous Matrices*

For this investigation, aqueous samples will be collected and submitted to ARI for analysis of Organophosphorus Pesticides, and Herbicides. Two 500ml Amber glass jars will be used per analyses. When collecting for duplicates, one more Amber glass jar will be required per each analysis.

### 8.2.3 *Sample Bottle decontamination*

Sample bottles will be decontaminated in accordance with the USEPA document "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, December 1992".

## 8.3 **Sample Handling, Packaging and Shipment**

The sampling team will assist the ENTRIX and/or CRA Field Supervisor with the preparation of samples being run for Organophosphorous Pesticides and Herbicides to be shipped to ARI. Following sample collection, the exterior of the sample containers will be decontaminated near the sampling location. Sample documentation and packaging will be performed in accordance with the procedures outlined in A Compendium of Superfund Field Operations Methods (USEPA, December 1987). Samples will be packaged for shipment as outlined in the FSP.

## 9.0 **Organic Free Water**

Organic free water will be used at the site as the final rinse during the decontamination of equipment. It will also be used when collecting equipment blanks for analysis at the laboratory. Organic free water is available through the laboratory and will be received from them when necessary. However, because of the volume of water that will be needed for a project as large as this, a water purification system that will produce organic free water will be put into place. This purification system will contain a reverse osmosis system on its front end and will get the water supply intake from available potable water. The water will then flow through the system making its final pass through an organic free filter/cartridge before being made available for use. Organic free water will be stored in clean glass, TeflonR, or stainless steel containers. QC objectives will be set up according to the manufacturer's specifications as well as industry standards. One sample per week will be taken and sent to STL to be analyzed for the project specific volatile compounds, and compared to the project objectives for volatiles, to keep the water quality in check. A sample will also be taken with the changing of the cartridge. Due to the mobile nature of volatile organic compounds, the project specific Method 8260 list will be analyzed. A log will be developed to

monitor the system and note any service checks. A service plan will be implemented according to manufacturer's specifications to proactively take care of issues before they become problems and keep the system running properly.

## **10.0 Update to Distribution List**

The address for EcoChem, Inc. has changed. The new and updated address is the following:

Eric Strout

EcoChem, Inc.

Dexter Horton Building

710 Second Ave., Suite 660

Seattle, WA 98104

(206) 233-9332

[estROUT@ecochem.net](mailto:estROUT@ecochem.net)

Number of Copies - 1

## **TABLES**

**Table 1**  
**Ecological Benchmarks, Method Detection Limits, and Reporting Limits for Organophosphate Pesticides in an Aqueous Matrix**

Organophosphate Pesticides Method SW846 8270D	CAS Number#	Method Detection Limit <sup>5</sup>	Reporting Limit	Ecological Screening Value <sup>1</sup>
		ug/L	ug/L	ug/L
Azinphos, methyl <sup>3</sup>	86-50-0	0.0647	1	0.01
Bidrin (dicrotophos) <sup>2</sup>	141-66-2	0.159	0.5	0.04
Bolstar <sup>2</sup>	35400-43-2	0.00141	0.2	0.15
Chlorpyrifos <sup>3</sup>	2921-88-2	0.00397	0.04	0.04
Chlorfenvinphos <sup>2</sup>	470-90-6	0.00818	0.2	0.04
Ciodrin <sup>2</sup>	7700-17-6	0.01423	0.2	0.66
Coumaphos <sup>2</sup>	56-72-4	0.00826	0.2	15
Demeton <sup>3</sup>	8065-48-3	0.0022	0.2	0.1
Diazinon <sup>3</sup>	333-41-5	0.00546	0.04	0.17
Dichlorvos <sup>2,6</sup>	62-73-7	0.00954	0.08	0.006
Dimethoate <sup>2</sup>	60-51-5	0.00307	0.2	0.2
Disulfoton <sup>4</sup>	298-04-4	0.00335	0.04	0.037
Epn (ent) <sup>2</sup>	2104-64-5	0.00561	0.2	0.01
Ethion <sup>2</sup>	563-12-2	0.00689	0.2	0.006
Ethoprop <sup>2</sup>	13194-48-4	0.00564	0.2	0.44
Fensulfothion <sup>2</sup>	115-90-2	0.0646	0.5	1
Fenthion <sup>2</sup>	55-38-9	0.0048	0.2	0.04
Malathion <sup>3</sup>	121-75-5	0.00628	0.2	0.1
Merphos <sup>2</sup>	150-50-5	0.267	1	130
Methyl Parathion <sup>4</sup>	298-00-0	0.00154	0.2	0.02
Mevinphos <sup>2</sup>	7786-34-7	0.00379	0.04	0.016
Naled <sup>2,6</sup>	300-76-5	0.00954	0.08	0.036
Parathion (niran) <sup>3</sup>	56-38-2	0.0039	0.2	0.013
Phorate <sup>4</sup>	298-02-2	0.00239	0.04	0.21
Ronnel <sup>2</sup>	299-84-3	0.00352	0.2	0.18
Stirofos <sup>2</sup>	22248-79-9	0.00289	0.2	0.19
Thiodiphosphoric acid	3689-24-5	0.04	0.2	0.023
Tokuthion <sup>2</sup>	3464-346-4	0.00623	0.2	0.13

Note: These screening values are preliminary and are subject to modification, compilation, and review of additional information.

AWQC - Ambient water quality criterion

EC50 - Concentration at which an effect was observed to 50% of test organisms.

LC50 - Concentration lethal 50% of test organisms.

LOEC - Lowest observed effect concentration

NA - Not available - data have not yet been identified.

OPP - USEPA Office of Pesticide Programs

<sup>1</sup> Screening values were derived by applying the following uncertainty factors to study endpoints:

LC50,EC50 - 10

LOEC - 5

(above ratios were based on conservative acute:chronic and LOEC:NOEC ratios using available data)

<sup>2</sup> USEPA AQUIRE database - freshwater organisms (<http://www.epa.gov/ecotox/>)

<sup>3</sup> USEPA Ambient Water Quality Criterion for the protection of aquatic life - chronic

<sup>4</sup> From USGS 2006. Pesticides in the Nations Streams and Groundwater, 1992-2001. Circular 1291.

Derived from USEPA Reregistration Eligibility Decisions and ecological risk assessments from the Office of Pesticide Programs.

<sup>5</sup> Method Detection Limits based on Analytical Resources, Inc. results.

<sup>6</sup> Dichlorvos and Naled are coeluting compounds.

ug/L - micrograms per Liter

**Table 2**  
**Ecological Benchmarks, Method Detection Limits and Reporting Limits for Organophosphate Pesticides in a Solid Matrix**

Analyte	CAS#	Method Detection Limits <sup>3</sup>	Reporting Limits	Ecological Screening Value
		ug/Kg	ug/Kg	ug/Kg
Azinphos, methyl <sup>1</sup>	86-50-0	17.8	100	32000
Bidrin (dicrotophos)	141-66-2	2.51	20	NA
Bolstar	35400-43-2	0.94	20	NA
Chlorpyrifos <sup>2</sup>	2921-88-2	1.26	4	10
Chlorfenvinphos	470-90-6	3.89	20	NA
Ciodrin	7700-17-6	1.65	20	NA
Coumaphos	56-72-4	1.26	20	NA
Demeton	8065-48-3	3.66	20	NA
Diazinon <sup>1</sup>	333-41-5	1.94	4	270
Dichlorvos <sup>4</sup>	62-73-7	3.42	8	NA
Dimethoate <sup>2</sup>	60-51-5	1.04	20	218
Disulfoton	298-04-4	2.5	4	NA
Epn (ent)	2104-64-5	0.86	20	NA
Ethion	563-12-2	0.93	20	NA
Ethoprop	13194-48-4	1.56	20	NA
Fensulfothion <sup>1</sup>	115-90-2	16.3	50	51
Fenthion	55-38-9	3.76	20	NA
Malathion <sup>2</sup>	121-75-5	1.33	20	100000
Merphos	150-50-5	75.9	100	NA
Methyl Parathion	298-00-0	0.92	20	NA
Mevinphos	7786-34-7	1.72	4	NA
Naled <sup>4</sup>	300-76-5	3.42	8	NA
Parathion (niran) <sup>2</sup>	56-38-2	0.88	20	0.34
Phorate <sup>2</sup>	298-02-2	4.78	4	0.496
Ronnel	299-84-3	1.6	20	NA
Stirofos	22248-79-9	0.87	20	NA
Thiodiphosphoric acid tetraethyl ester <sup>2</sup>	3689-24-5	4	20	596
Tokuthion	3464-346-4	1.87	20	NA

Note: These screening values are preliminary and are subject to modification following compilation and review of additional information.

NA - Not available - data have not yet been identified.

EC50 - Concentration at which an effect was observed to 50% of test organisms.

NOEC - No observed effect concentration

LETC - Lethal Threshold Concentration

Uncertainty factor of 10 was applied to chronic EC50 (35d *Folsomia candida* survival) to derive NOEC.

Uncertainty factor of 100 applied to LETC (28d *Lumbricus* mortality) and EC100 (7d *Allolobophora caliginosa* weight)

<sup>1</sup>USEPA ECOTOX Database (<http://www.epa.gov/ecotox>)

<sup>2</sup>USEPA Region 5 Ecological Screening Levels (<http://www.epa.gov/Region5/rcraca/edql.htm>)

<sup>3</sup>Method Detection Limits based on Analytical Resources, Inc. results.

<sup>4</sup>Dichlorvos and Naled are coeluting compounds.

ug/Kg - micrograms per Kilogram

**Table 3**  
**Ecological Benchmarks, Method Detection Limits, and Reporting Limits for Herbicides in Aqueous Matrices**

Herbicides SW846 Method 8151	CAS Number	Method Detection Limit <sup>3</sup>	Reporting Limit	Ecological Screening Value <sup>1</sup>
		ug/L	ug/L	ug/L
2,4-D (2,4-dichlorophenoxyacetic acid) <sup>2</sup>	86-50-0	0.079	1	2
2,4-DB (2,4-dichlorophenoxy butyric acid) <sup>2</sup>	141-66-2	0.27	5	20
2,4,5-T (2,4,5-trichlorophenoxyacetic acid) <sup>2</sup>	35400-43-2	0.013	0.25	0.13
2,4,5-TP (2,4,5-trichlorophenoxy propionic acid) <sup>2</sup>	2921-88-2	0.069	0.25	3.4
Alachlor <sup>4</sup>	470-90-6	40	200	0.016

Note: These screening values are preliminary and are subject to modification compilation and review of additional information.

<sup>1</sup>For Herbicides - Screening values were derived by applying the following uncertainty factors to study endpoints:

LC50 - 100

LOEC - 10

NOEC - 1

Where LC50 = Concentration lethal 50% of test organisms; LOEC = Lowest observed effect concentration

<sup>2</sup> USEPA AQUIRE database - freshwater organisms (<http://www.epa.gov/ecotox/>)

<sup>3</sup>Method Detection Limits based on Analytical Resources, Inc. results.

<sup>4</sup>Reported value based on 75% recovery of a 400 ng/L Alachlor spike and 75% recovery of a 100 ng/L spike.

ug/L - micrograms per Liter

**Table 4**  
**Ecological Benchmarks, Method Detection Limits, and Reporting Limits for Herbicides in a Solid Matrix**

Herbicides SW846 Method 8151	CAS Number#	Method Detection Limit <sup>3</sup>	Reporting Limit	Ecological Screening Value <sup>1</sup>
		ug/Kg	ug/Kg	ug/Kg
2,4-D (2,4-dichlorophenoxyacetic acid)	86-50-0	13.8	33	NA
2,4-DB (2,4-dichlorophenoxy butyric acid)	141-66-2	44.2	170	NA
2,4,5-T (2,4,5-trichlorophenoxyacetic acid) <sup>2</sup>	35400-43-2	2.72	8.3	596
2,4,5-TP (2,4,5-trichlorophenoxy propionic acid)	2921-88-2	3.41	8.3	NA
Alachlor <sup>4</sup>	470-90-6	4	2	NA

Note: These screening values are preliminary and are subject to modification compilation and review of additional information.

<sup>1</sup>For Herbicides - Screening values were derived by applying the following uncertainty factors to study endpoints:

LC50 - 100

LOEC - 10

NOEC - 1

Where LC50 = Concentration lethal 50% of test organisms; LOEC = Lowest observed effect concentration

<sup>2</sup> From USGS 2006. Pesticides in the Nations Streams and Groundwater, 1992-2001. Circular 1291.

Derived from USEPA Reregistration Eligibility Decisions and ecological risk assessments from the Office of Pesticide Programs.

<sup>3</sup>Method Detection Limits based on Analytical Resources, Inc. results.

ug/Kg - micrograms per Kilogram

<sup>4</sup>Reported value based on 75% recovery of a 400 ng/L Alachlor spike and 75% recovery of a 100 ng/L spike.

Table 5

## Ecological Benchmarks, Method Detection Limits and Reporting Limits for Organophosphate Pesticides in Sediment

Analyte	CAS#	Method Detection Limit <sup>3</sup>	Reporting Limit	Ecological Screening Value
		ug/Kg	ug/Kg	ug/Kg
Azinphos, methyl <sup>1,8</sup>	86-50-0	17.8	100	0.088
Bidrin (dicrotophos) <sup>7</sup>	141-66-2	2.51	20	0.147
Bolstar <sup>8</sup>	35400-43-2	0.94	20	0.001
Chlorpyrifos <sup>2,7</sup>	2921-88-2	1.26	4	2.732
Chlorfenvinphos <sup>12</sup>	470-90-6	3.89	20	0.236
Ciodrin	7700-17-6	1.65	20	NA
Coumaphos <sup>9</sup>	56-72-4	1.26	20	13.845
Demeton	8065-48-3	3.66	20	NA
Diazinon <sup>1,6</sup>	333-41-5	1.94	4	1.9
Dichlorvos <sup>4,7</sup>	62-73-7	3.42	8	0.002
Dimethoate <sup>2,6</sup>	60-51-5	1.04	20	N/A
Disulfoton <sup>6</sup>	298-04-4	2.5	4	324
Epn (ent) <sup>10</sup>	2104-64-5	0.86	20	0.4
Ethion <sup>7</sup>	563-12-2	0.93	20	0.792
Ethoprop <sup>8</sup>	13194-48-4	1.56	20	0.708
Fensulfothion <sup>2,8</sup>	115-90-2	16.3	50	0.0715
Fenthion <sup>8</sup>	55-38-9	3.76	20	0.748
Malathion <sup>2,6</sup>	121-75-5	1.33	20	0.67
Merphos <sup>7</sup>	150-50-5	75.9	100	81120
Methyl Parathion <sup>6</sup>	298-00-0	0.92	20	N/A
Mevinphos <sup>8</sup>	7786-34-7	1.72	4	0.0002
Naled <sup>4,7</sup>	300-76-5	3.42	8	0.034
Parathion (niran) <sup>2,6</sup>	56-38-2	0.88	20	0.757
Phorate <sup>3,6</sup>	298-02-2	4.78	4	N/A
Ronnel <sup>7</sup>	299-84-3	1.6	20	3.614
Stirofos <sup>7</sup>	22248-79-9	0.87	20	0.546
Thiodiphosphoric acid tetraethyl ester <sup>2,5</sup>	3689-24-5	4	20	N/A
Tokuthion <sup>11</sup>	3464-346-4	1.87	20	0.01

Note: These screening values are preliminary and are subject to modification following compilation and review of additional information.

NA - Not available - data have not yet been identified.

EC50 - Concentration at which an effect was observed to 50% of test organisms.

NOEC - No observed effect concentration

LETC - Lethal Threshold Concentration

Uncertainty factor of 10 was applied to chronic EC50 (35d *Folsomia candida* survival) to derive NOEC.

Uncertainty factor of 100 applied to LETC (28d *Lumbricus* mortality) and EC100 (7d *Allolobophora caliginosa* weight)

<sup>1</sup> - USEPA ECOTOX Database (<http://www.epa.gov/ecotox>)

<sup>2</sup> - USEPA Region 5 Ecological Screening Levels (<http://www.epa.gov/Region5/rcraca/edql.htm>)

<sup>3</sup> Method Detection Limits based on Analytical Resources, Inc. results.

<sup>4</sup> Dichlorvos and Naled are coeluting compounds.

<sup>5</sup> Values are performance based on 75% recovery of a 400 ng/L Alachor spike and 75% of a 100 ng/L spike. More detailed study in progress.



## Table 5

### Ecological Benchmarks, Method Detection Limits and Reporting Limits for Organophosphate Pesticides in Sediment

Note: Sediment ESV calculated by Equilibrium Partitioning Method, assuming 1% as the sediment organic content and using:

<sup>6</sup>Water Quality Criteria as specified in IEPA Agency Comments on the Quality Assurance Project Plan Addendum, June, 2006 (IEPA, August, 2006).

<sup>7</sup>Organic Carbon Partitioning Coefficient values based upon numbers found in the Risk Assessment Information System (RAIS) database.

<sup>8</sup>S. Orme and S. Kegley, PAN Pesticide Database, Pesticide Action Network (<http://www.pesticideinfo.org>).

<sup>9</sup>USEPA Office of Pesticides and Toxic Substances Memorandum Subject: *Revised Tier 1 Drinking Water Assessment for Coumaphos*.

<sup>10</sup>USDA ARS Pesticide Properties Database (May, 1995).

<sup>11</sup>Texas Commission on Environmental Quality (TCEQ) Table for 1993 Risk Reduction Rule (March 31, 2006).

<sup>12</sup>Syracuse Research Corporation (SRC) ([www.syrres.com](http://www.syrres.com))

ug/Kg - micrograms per Kilogram

**Table 6**  
**Ecological Benchmarks, Method Detection Limits, and Reporting Limits for Herbicides in Sediment**

Herbicides SW846 Method 8151	CAS Number#	Method Detection Limit <sup>2</sup>	Reporting Limit	Ecological Screening Value <sup>1</sup>
		ug/Kg	ug/Kg	ug/Kg
2,4-D (2,4-dichlorophenoxyacetic acid) <sup>3</sup>	86-50-0	13.8	33	0.9
2,4-DB (2,4-dichlorophenoxy butyric acid) <sup>4</sup>	141-66-2	44.2	170	22
2,4,5-T (2,4,5-trichlorophenoxyacetic acid) <sup>4</sup>	35400-43-2	2.72	8.3	0.36
2,4,5-TP (2,4,5-trichlorophenoxy propionic acid) <sup>5</sup>	2921-88-2	3.41	8.3	6.5
Alachlor <sup>4</sup>	470-90-6	4.0	2.0	68

Note: These screening values are preliminary and are subject to modification compilation and review of additional information.

<sup>1</sup>For Herbicides - Water quality values were derived by applying the following uncertainty factors to study end points:

LC50 - 100

LOEC - 10

NOEC - 1

Where LC50 = Concentration lethal 50% of test organisms; LOEC = Lowest observed effect concentration

and using the USEPA AQUIRE database - freshwater organisms (<http://www.epa.gov/ecotox/>) as well as USGS Pesticides in the Nations Streams and Groundwater, 1992-2001

Circular 1291. Derived from USEPA Reregistration Eligibility Decisions and ecological risk assessments from the Office of Pesticide Programs.

<sup>2</sup>Method Detection Limits based on Analytical Resources, Inc. results.

ug/Kg - micrograms per Kilogram

Note: Sediment ESV calculated by Equilibrium Partitioning Method, assuming 1% as the sediment organic content and using water quality values derived from the above noted method.

<sup>3</sup>S. Orme and S. Kegley, PAN Pesticide Database, Pesticide Action Network (<http://www.pesticideinfo.org>).

<sup>4</sup>USDA ARS Pesticide Properties Database (May, 1995).

<sup>5</sup>USEPA Office of Pesticides and Toxic Substances Memorandum Subject: *Revised Tier 1 Drinking Water Assessment for Coumaphos*.

**APPENDIX A**  
**LABORATORY SOPs**



**Analytical Resources, Incorporated**  
Analytical Chemists and Consultants

**Standard Operating Procedure**  
**Organophosphorous Pesticide Analysis**  
**EPA Method 8270D (GC-MS-SIM)**

**SOP 805S**  
**Revision 001**

**Revision Date: 8/25/06**  
**Effective Date: 8/25/06**

Prepared By:

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Approvals:

Jan N. Zifer  
Laboratory / Section Manager

David R. Mitchell  
Quality Assurance





## GC-MS Analysis – Selective Ion Monitoring (SIM)

### 1. Scope and Application

- 1.1. This SOP describes the use of gas chromatography/mass spectrometry (GC/MS) using selective ion monitoring (SIM) to determine the concentration Organophosphorous pesticide compounds listed in Appendix 20.2 in extracts prepared from various types of solid waste matrices, soils, tissues, sediments and waters. Some text has been taken directly from the analytical methods referenced in Section 19
- 1.2. Routine reporting limits (RL) for each compound are listed in Appendix 20.2. RLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector and/or display chromatographic interference.
- 1.3. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

### 2. Summary of Procedure

- 2.1. An measured amount of sample, normally 500 mL for aqueous samples and 7.5 grams for solid samples) is extracted by a matrix appropriate technique. Sample extraction is continuous liquid-liquid extraction or separatory funnel extraction for aqueous samples and sonication for solid samples. Section 1.1 lists other sample matrices and extraction methods amenable to analysis using this method. The extracted sample may be subjected to an appropriate cleanup and concentrated as described in ARI's sample preparation SOPs. Routine final volumes are 0.5 mL for aqueous samples and either 0.5 or 1 mL for solid samples.
- 2.2. Sample extracts are analyzed using GC-MS with the MS in the SIM mode. Prior to analysis the GC/MS system is tuned and calibrated as described later in this document. Target analytes are identified by comparing their chromatographic retention times and mass spectra with known standards. Detected analytes are quantified by comparing their mass spectral response with that of standards of known concentrations relative to an internal standard.

### 3. Definitions

- 3.1. Analytical Batch-
- 3.2. DDT Breakdown Factor- the ratio of the sum of the areas of the DDT breakdown products (DDD and DDE) divided by the summated areas of DDT and the DDT breakdown products. The factor is used to measure injector inertness.
- 3.3. DFTTP (decafluorotriphenylphosphine)



- 3.4. Extracted Ion Current Profile (EICP) - plot of the abundance of a specific ion as a function of time.
- 3.5. Laboratory Control Sample is a volume of clean reference matrix (reagent water for water samples, or sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The LCS is spiked with all surrogates and target analytes required by the method.
- 3.6. Laboratory Information Management System (LIMS)
- 3.7. Limit of detection (LOD)
- 3.8. Method Blank: a volume of clean reference matrix (reagent water for water samples, or sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.
- 3.9. Matrix Duplicates are replicate aliquots of the same sample taken through the entire analytical procedure Matrix Spike Analysis
- 3.10. Peak Tailing Factor- A measure of the relative activity / inertness of the chromatographic system as defined in Appendix 20.6.
- 3.11. Reconstructed Ion Current (RIC) - plot of the total instrument response versus time.
- 3.12. Relative Retention Time (RRT) - the elution time of an analyte relative to the elution time of its associated internal standard.
- 3.13. Sample Deliver Group
- 3.14. Scan Descriptor- the summary of masses and scan time per mass the mass spectrometer uses through the chromatographic run.
- 3.15. Second Source Standard
- 3.16. Selected Ion Monitoring (SIM) - a mode of data acquisition wherein the mass spectrometer is programmed to scan for a limited number of specific masses, increasing the amount of time spent searching for each of these masses.
- 3.17. Standard Reference Material (SRM) or Certified Reference Material (CRM)
- 3.18. Surrogate Standards are compounds chemically similar to target analytes that are added to a sample prior to the extraction process. The recovery of surrogates is used to evaluate extraction efficiency. Surrogates are halogenated or isotope labeled compounds not expected to be detected in environmental samples that are added to every sample analyzed.

#### **4. Interferences**

- 4.1. All GC/MS data (method blanks, samples, and spikes) must be evaluated for interferences.

Analysts determine the source of interference and generate a Corrective Action Issue.



4.2. Contamination by sample to sample carryover may occur whenever high-concentration and low-concentration samples are analyzed sequentially. The autosampler syringe is rinsed with solvent between samples to reduce possible carryover. It is prudent to follow an unusually concentrated sample with an analysis of an instrument blank to check for cross contamination.

## 5. Safety

5.1. The toxicity and carcinogenicity of sample extracts and calibration standards used in this method are not precisely defined. Treat extracts and standards as health hazards, and reduce exposure to them as much as possible.

5.1.1. Skin contact with all chemicals should be minimized by the used of nitrile gloves, safety glasses, and laboratory coats.

5.1.2. All sample extracts and standard solutions must be handled in a fume hood to avoid exposure to fumes.

5.2. ARI maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Consult MSDS sheets for specific safety information prior to handling a solvent or reagent.

## 6. Equipment and Supplies

6.1. Gas chromatograph/mass spectrometer system

6.1.1. Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, autosampler, and gases. The capillary column should be directly coupled to the source of the mass spectrometer.

6.1.2. Fused-silica capillary column - 30 m x 0.32 mm ID (or 0.25 mm ID) 0.25 -0.5  $\mu\text{m}$  film thickness coated with cross bonded 5% diphenyl/95% dimethyl polysiloxane stationary phase (Phenomonex ZB-5 or equivalent).

6.1.3. Mass spectrometer - Capable of scanning from 35 to 500 amu in 1 second or less. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Appendix 20.3 when 1-2  $\mu\text{L}$  of the GC/MS tuning standard is injected onto the GC (25-50 ng of DFTPP).

6.1.4. GC/MS interface – ARI uses capillary-direct into the mass spectrometer source interface.





6.2. Data system - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.2.1. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

6.2.2. A mass spectral library utilizing a specific Organophosphorous Pesticide scan descriptor must be created to provide library search information for automated compound identification and data reduction. The mass spectral library should be created using the midpoint of the initial calibration. The identical Organophosphorous Pesticide scan descriptor must be employed for the initial calibration and all subsequent analyses in order for such comparisons to remain valid, otherwise a new mass spectral library must be created.

6.2.3. The data will be securely stored for at least seven years from date of acquisition.

6.3. Syringe - 10  $\mu$ L, 25  $\mu$ L, 50  $\mu$ L, 100  $\mu$ L, 500  $\mu$ L, 1000  $\mu$ L

6.4. Volumetric flasks, Class A - 10 mL to 1000 mL.

6.5. Bottles - glass with Teflon-lined screw caps or crimp tops.

6.6. Balance - Analytical, 0.0001 g

6.7. Autosampler vials- amber 2 ml autosampler vials with crimp caps.

6.8. Derivatization apparatus and reagents

6.8.1. A 40 mL VOA vial, with cap and Teflon lined septum.

6.8.2. A Teflon tube, pushed through the septum

6.8.3. Diazald

6.8.4. Methanol

6.8.5. 3M NaOH in methanol

## 7. Reagents and Standards

7.1. Organic Solvents

7.1.1. High purity, reagent grade solvents and chemicals are used in all analyses.

7.1.2. The laboratory stocks acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, methanol and other appropriate solvents for preparing standards.

7.2. Spiking and calibration standards are listed in Section 10. The standard solution are prepared using procedures outlined in ARI SOP 1012S.



## 8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts are delivered to Refrigerator 17 in the instrument laboratory by extractions technicians.

The extracts are signed into the GC laboratory Extract Refrigerator Log when delivered.

8.2. Analysts in the instrument lab assume custody of the sample extracts and then move them into Refrigerator 15.

8.3. Extracts must be stored at  $4 \pm 2$  protected from light.

8.4. Extracts must be analyzed within 40 days of the first day of extraction.

9. **Quality Control** procedures include negative and positive controls, verification of detection limits, calibration and method selectivity.

### 9.1. Negative Control

9.1.1. Method blanks (MB) are used to assess the preparation batch for possible contamination resulting from the sample preparation process. extraction and analysis are performed as follows:

9.1.2. One MB is analyzed with every preparation batch of twenty or fewer samples.

9.1.3. The MB is analyzed once for each preparation batch unless MB data for each instrument is mandated by project-specific requirements a fully validatable data package is required.

9.1.4. When post preparation contamination is suspected, the method blank will be reanalyzed in a manner that will verify the source of contamination.

9.1.5. An aqueous MB is prepared using 0.5 or 1.0 Liters of Type 1 water from ARI's centralized water purification system. Method blanks for solid samples (soil and sediment), are prepared using 1 to 100 g purified sodium sulfate.

9.1.6. Method blanks must be processed in exactly the same manner as other samples in a preparation batch.

### 9.2. Positive Controls

9.2.1. LCS analyses aid in the evaluation of the total analytical process including all preparation and analytical procedures.

9.2.1.1. Recovery of compounds added to the LCS are compared to established statistical criteria calculated from historic analytical data as described in Section 15.5

9.2.1.2. An LCS sample is analyzed with every batch of twenty of fewer samples.

9.2.1.3. When there is not sufficient sample to analyze an MSD and/or MSD, an LCSD may be analyzed, at the client's discretion, and compared to the LCS to evaluate method precision.

9.2.2. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) analyses indicate the effect of the sample matrix on the precision and accuracy of the results using the selected method. MS



and MSD results are not normally used to determine the validity of the entire batch of analyses.

9.2.2.1. Each preparatory batch of twenty or less samples of a similar sample matrix must include both MS and MSD analyses

9.2.2.2. MS samples are spiked with all of the target analytes as indicated in Appendix 20.2.

9.2.2.3. MS and MSD results are evaluated against statistical control limits calculated from ARI's historical data. Project or QA protocol specific control limits may substitute for ARI's calculated values.

9.2.2.4. QA/QC samples (rinsate blank, trip blank, SRM, etc.) submitted by a client must not be used to prepare MS samples.

9.2.2.5. When ARI's client designates a specific sample for MS/MSD analysis, that sample must be used. When there is insufficient sample to perform MS/MSD analyses ARI will immediately inform the client. The client will approve one of the following options:

9.2.2.5.1. MS/MSD analyses are deleted from the sample batch.

9.2.2.5.2. An alternate sample is designated for MS/MSD analyses

9.2.2.5.3. LCS/LCSD analyses are used to assess method precision.

9.2.2.6. The client's rationale and decision must be documented on the "Analytical Notes" form and reported in the case narrative.

9.2.2.7. Dilution of MS/MSD extracts to get either spiked compounds or native analytes on scale is not required.

9.2.3. Matrix Duplicates (MD) may be analyzed as required by a specific client request and/or QA protocol. MD analyses are used to assess the precision of analytical results in a given sample matrix. The results of an MD analyses are normally expressed as an RPD (relative percent difference) value.

9.2.4. Surrogates Standards (SS) are added to all samples prior to extraction to provide a measure of method efficiency (recovery) for each sample matrix.

9.2.4.1. SS are added to all samples including MB, LCS and MS.

9.2.4.2. SS are chosen to represent the various chemistries of the target analytes. ARI's routine SS are noted in Appendices 20.2.

9.2.5. The reporting limits for analytes in this procedure are defined by the lowest concentration calibration standard. MDL studies, as described in ARI SOP 1018S, are conducted to determine an LOD. An analyte concentration between the RL and the LOD (MDL) may be reported but must be flagged to indicate the higher analytical uncertainty associated with these values in this range.



9.2.6. Data reduction procedures are standardized and documented in SOPs

9.2.7. Method selectivity is evaluated using the following checks:

9.2.7.1. Prior to any analysis each mass spectrometer must be hardware-tuned to meet the criteria in Appendix 20.3.

9.2.7.1.1. Inject 25-50 ng/uL solution of DFTPP, p,p'-DDT, benzidine and pentachlorophenol.

9.2.7.1.1.1. Evaluate the ion abundance of DFTPP using one of the following scan scenarios:

9.2.7.1.1.1.1. one scan at the peak apex

9.2.7.1.1.1.2. use the mean of the apex and the preceding and following scans

9.2.7.1.1.1.3. use the average across the entire peak.

9.2.7.1.1.2. Background correction should be compliant with method specifications and employed only for the purpose of correcting for instrument background ions. Do not subtract part of the DFTPP peak.

9.2.7.1.1.3. Degradation of DDT to DDE and DDD should not exceed 20%. DDT percent breakdown is calculated by dividing the sum of the DDD and DDE areas by the sum of the areas of DDT, DDE and DDD and then multiplying this result by 100.

9.2.7.1.1.4. Benzidine and pentachlorophenol should be present at their normal responses, with no visible peak tailing. Peak tailing factors are calculated using the formula in Appendix 20.6. Tailing factors must be <3.0 for benzidine and <5.0 for pentachlorophenol. Non-compliances must be documented on the "Analyst's Notes" form and reported in the case narrative.

9.2.7.1.2. Section 16 includes procedures to bring the instrument into compliance for tuning, DDT breakdown, or peak tailing factors.

9.2.7.2. The relative retention time (RRT) of each target analyte must vary be less than 0.06 RRT units in the calibration standards. Late-eluting target analytes usually have much better agreement. The RRT is calculated by dividing the retention time of the target analyte/surrogate by the retention time of its assigned internal standard.

9.2.7.3. The internal standards selected should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Appendix 20.2). If interferences are noted, use the next most intense ion as the quantitation ion.



9.2.7.4. Internal Standard Response (EICP) area and retention time data are to be evaluated during and/or immediately after the analysis. The response for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis. The retention time shift for each of the internal standards must be within  $\pm 0.50$  minutes (30 seconds) between the sample and the most recent continuing calibration analysis.

9.2.7.5. Statistical Control- Internal quality control limits for analyte spike and surrogate recoveries and relative percent difference for matrix spike and matrix spike duplicates are statistically generated on a periodic basis. These quality control limits are provided to bench chemists, managers, and QA staff as tools for assessing data quality in real-time at the point of data generation. Practical considerations relating to their dynamic nature require their presentation in a document separate from this SOP. All analysts using this SOP must use it in conjunction with Control Limit documentation in order to assess data quality and any possible need for corrective actions. Current control limits may be found in the ARI QAP.

9.2.7.6. Initial Demonstration of Proficiency- Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination performed, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat these procedures whenever new staff is trained or significant changes in instrumentation or procedure are made. See Methods 8000 and 3500 for information on how to accomplish this demonstration.

## 10. Calibration and Standardization

10.1. Calibration standards - A minimum of six calibration standards are used. The lowest concentration calibration standard defines the reporting limit and the others should correspond to the range of concentrations found in real samples but must not exceed the linear range of the GC/MS system. The lowest calibration point of each individual target analyte becomes the reporting limit for that target. All target analytes quantitated below the lowest calibration point for any target analyte must be qualified with a "J" flag to show the quantitation is below the working range of the curve. Calibration standards are prepared from the working calibration solutions at concentrations of 0.04, 0.20, 0.50, 1.0, 5.0, 10, and 20  $\mu\text{g}/\text{mL}$  for each of the target analytes and surrogates. The calibration standards are made with a final volume of 0.5 ml in an amber vial with a crimp cap. 10  $\mu\text{L}$  internal standard solution is added for each 500  $\mu\text{L}$  of extract prior to analysis. Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table 1 may be included). However, no target analyte may be quantitated



without first being calibrated. All standards should be stored at 4°C and should be freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard should be prepared weekly with the expiration date and standard identifications on the label and stored at 4°C. If the analyst suspects degradation the standard should be replaced.

10.2. If the DFTPP tuning and peak tailing factors meet acceptance criteria, the initial calibration may proceed by injecting the calibration standards prepared as described in Section 10.1 into the analytical column.

### 10.3. Calibration Requirements

10.3.1. The average RRF should be calculated for each compound. The percent relative standard deviation ( $\% \text{RSD} = 100 [\text{SD}/\text{average RRF}]$ ) should also be calculated for each compound. The % RSD should be less than 15% for each compound, in which case the average response factor will be used for quantitation as it is constant over the calibration range. Target analytes that exceed 15% RSD will utilize an alternative calibration option. If the analyst determines that there is no detector or chromatographic peak saturation and that the chromatographic system is functioning properly, then the analyst will proceed as follows:

10.3.1.1. A linear fit calibration that does not include the origin and generates a coefficient of determination ( $R^2$ ) that is greater than or equal to 0.99 is acceptable. ARI will attempt to force calibration curve through zero. Forcing the curve through zero is not the same as including the origin as a point in the calibration. When the curve is forced through zero, the intercept is set to 0 before the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom.

10.3.1.2. Next the analyst may attempt a quadratic non-linear calibration which may be used with target analytes that have six calibration points only. An additional initial calibration point may be considered for target analytes that require non-linear calibration if only five calibration points are available.

10.3.1.3. Individual targets that are unable to meet the above requirements must be documented in the case narrative. The initial calibration may still be valid if approved on an individual basis by the Laboratory Supervisor and/or the Laboratory Manager.

10.3.1.4. See EPA Method 8000C Section 11 for reference to linear fit and non-linear (quadratic) calibration.

10.3.1.5. Individual targets that are unable to meet the requirements in Section 10.3.1 must be documented in the case narrative. The initial calibration may still be valid if approved on an individual basis by the Laboratory Supervisor, and/or the Laboratory Manager.



10.4. Calibration curve verification - Prior to use for sample analysis, the acceptability of a calibration curve must be verified through analysis of an initial calibration verification standard (ICV) obtained from a second source. The ICV must be derived from a different source than the stock standard used to prepare the calibration curve. Stock standards from different lots supplied by the same manufacturer may be used to satisfy the independent source requirement. The ICV should show 70%-130% recovery for all compounds when compared to the initial calibration curve. Any analytes exceeding 70%-130% recovery will be documented in the analysts notes provided with the initial calibration.

10.5. Using data from the mid-point of the initial calibration, verify that the reference mass spectrum library accurately identifies all target analytes.

10.6. Daily GC/MS calibration verification - Performed at the beginning of each 12-hour analytical shift.

10.6.1. Prior to analysis of samples, the GC/MS tuning standard must be analyzed using a full scan of 35-500 amu. A 25-50 ng/uL injection of DFTPP must result in a mass spectrum for DFTPP which meets the specified criteria (Appendix 20.3). These criteria must be demonstrated during each 12 hour shift. The 12 hour shift begins from when the DFTPP was injected and does not include time of elution. As discussed in Section 9.2.7.1.1.3 the DFTPP check should also measure injector port inertness through the evaluation of DDT breakdown and the calculation of peak tailing factors for pentachlorophenol and benzidine.

10.6.2. Analysis of a continuing calibration verification standard (CCV) at mid-concentration (2.5/12.5 ng/uL), containing each compound of interest, including all required surrogates, must be performed once every 12 hours before sample analysis, using the introduction technique used for the initial calibration. The results from the CCV analysis must meet the acceptance criteria detailed below in Sections 10.6.2.1 through 10.6.2.3.

10.6.2.1. Percent Difference Criteria- After the tune check has been made, check the validity of the calibration by calculating the %D for each analyte.

$\% \text{ Difference} = \frac{RRF_i - RRF_c}{RRF_i} * (100)$
Where:
RRFi = Average relative response factor from initial calibration.
RRFc = Relative response factor from current verification check standard.

10.6.2.2. The percent difference for each analyte should be less than 20%. However, up to one analyte may have a percent difference greater than 20%, so long as its percent



difference is less than 40%. If the percent difference criterion is not met corrective action must be taken. See Section 16.3 or procedures for dealing with CCV failure. This criterion must be met before sample analysis begins. All corrective actions must be recorded on an "Analyst Notes" form.

10.6.2.3. The internal standard responses and retention times in the CCV must be evaluated immediately after or during data acquisition. Relative retention times should remain constant and resolution between isomeric pairs must be maintained for any changes in retention times. If the retention time for any internal standard changes by more than 5 minutes or the response of any internal standard (EICP area) changes by a factor of two (-50% to +100%) from that in the mid-point standard level (2.5 ng/uL) of the most recent initial calibration sequence, then the system should be inspected for malfunctions and corrections made as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required only if mandated by project specific requirements.

## 11. Procedure

11.1. It is highly recommended that the extract be screened using GC/FID with the same type column used in the GC-MS. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds and may show high background samples that should be analyzed using a medium/high level extraction. The screening procedure is provided in Appendix 20.7.

11.2. Recommended GC/MS operating conditions are as tabulated in Appendix 20.4 (samples must be run using the same instrument conditions as the initial calibration).

11.3. Sample extracts are analyzed with the GC-MS the SIM mode. The instrument is toggled between the full scan and SIM modes using the Chemstation software.

11.4. Verify that the GC-MS method in use contains the proper scan descriptors for the target analytes. Scan descriptors are provided in Appendix 20.2.

11.5. Tune the GC-MS instrument as described in Section 9.2.7.1

11.6. Calibrate the instrument as described in Section 10

11.7. Spike each sample extract to be analyzed with internal standard solution using 2  $\mu$ L of the internal standard solution for every 0.1 mL of extract. (example: spike 10  $\mu$ L of internal standard stock into a 0.5 ml sample) This results in a concentration of 2 ng/ $\mu$ L of each internal standard

11.8. Prior to sample analysis a continuing calibration verification standard must be analyzed, and this CCV must meet the criteria found in Section 10.6. If time remains in the 12 hour QC period





begun with the initial calibration, the midpoint calibration standard from the initial calibration may be used as the CCV provided it meets the requirements found in Section 10.6.

- 11.9. Using the analyte RRT form the CCV to adjust the beginning of each scan group as necessary.
- 11.10. Enter all information required for the analytical batch in the instrument logbook.
- 11.11. Load the auto sampler and enter the sample sequence on the Target data system.
- 11.12. Print the run sequence and attach it to the instrument logbook
- 11.13. Begin GC/MS data acquisition
- 11.14. When the acquisition is complete, export the data files from the GC/MS data system to to the Target data system for data further analysis.
- 11.15. Process the data using the appropriate Target method.
- 11.16. Confirm that the computer has correctly identified all target analytes and properly performed all chromatographic integrations.
- 11.17. Perform necessary manual integrations following the guidelines in Appendix 20.1.
- 11.18. Document any manual integrations by printing a copy of magnified EICP signals and/or chromatograms showing manual integrations.
- 11.19. The analyst must sign and date all hardcopy data, especially that documenting manual integrations.
- 11.20. Export the quantitative numbers into ARI's LIMS where final reports will be generated.
- 11.21. Submit a hardcopy job file to management for review. The file will contain:
  - 11.21.1. Copies of all bracketing tune files
  - 11.21.2. Copies of all bracketing tune files CCVs
  - 11.21.3. Raw data for all the samples and associated QC analyses
  - 11.21.4. The LIMS report(s)
  - 11.21.5. An "Analyst's Notes" form that includes all deviations from normal routine procedures and any other noteworthy observations or events concerning sample analysis
- 11.22. All extracts should be stored in Refrigerator 15 in amber vials with teflon lined screw caps.

## 12. Data Analysis and Calculations

### 12.1. Qualitative Analysis

- 12.1.1. Target analytes are identified by comparing characteristic ions of the sample mass spectrum with that of a known reference standard. Reference mass spectra are generated by the laboratory using routine calibration data obtained using conditions identical to the sample analyses. Characteristic ions are either the three ions of greatest intensity, or any ions over 30% intensity relative to the base ion. Two criteria must be satisfied to verify identification:



- 12.1.1.1. The target analyte in the sample must elute within  $\pm 0.06$  RRT units of the RRT of the compounds in a calibration standard analyzed in the same 12 hour QC period.
- 12.1.1.1.1. When co-elution components prohibit accurate assignment of an RRT using total ion chromatogram, the RRT may be assigned by using extracted, ion-current profiles for ions unique to the component of interest.
- 12.1.1.2. The mass spectrum of the target analyte must correspond to that of the standard component.
- 12.1.1.2.1. Intensities of all characteristic ions must maximize in the same or an adjacent scan. A chromatographic peak selected by a data system based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time meets this criterion.
- 12.1.1.2.2. All ions present in the standard mass spectra at a relative intensity greater than 10% (the most abundant ion in the spectrum is equal to 100% intensity) must be present in the sample spectrum.
- 12.1.1.2.3. The relative intensities of ions specified in Section 12.1 in the standard and sample spectra must be within  $\pm 30\%$ . (Example: an ion with an abundance of 50% in the standard spectrum must have a corresponding ion between 20 and 80% in the sample spectrum.) Compounds with spectra not meeting this criterion may be considered acceptable and flagged with an M by an analyst or data (favors false positive results).
- 12.1.1.2.4. Structural isomers that produce similar mass spectra may be identified as individual isomers if they have sufficiently chromatographic resolution. Sufficient GC resolution is achieved when the height of the valley between two isomeric peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs and reported as the sum of both compounds by the analyst.
- 12.1.1.3. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be



met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

## 12.2. Quantitative analysis

12.2.1. Confirmed target analytes are quantified based on the integrated abundance from the EICP of the primary characteristic ion.

12.2.2. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of a given analyte.

12.2.3. When secondary ion quantitation is necessary due to interference, a short quantitation report list is generated. This quantitation contains the integrated areas of the affected compounds, based on the secondary ion(s) for that compound, and of the relevant internal standards. Identical reports must be generated for the sample with interference and for the relevant initial calibration. The report for the initial calibration is used to generate a relative response factor for the affected compound based on its secondary ion. This relative response factor is then used in the calculations for that compound in the affected sample. All quantitations using the primary characteristic ion use the average relative response factor from the initial calibration. The short quantitation report may be hand calculated by the analyst as long as it is signed and dated by the analyst.

12.2.4. Calculate the concentration of each identified analyte in the sample as follows:

<b>Concentration (µg/L) = <math>\frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_o)(V_i)}</math></b>
where:
$A_x$ = Area of characteristic ion for compound being measured
$I_s$ = Amount of internal standard injected (ng).
$V_t$ = Volume of total extract, taking into account dilutions (i.e. for a 1 to 10 dilution of a 1 ml extract $V_t = 10,000 \mu\text{L}$ )
$A_{is}$ = Area of characteristic ion for the internal standard
RRF = Relative response factor for compound being measured
$V_o$ = Volume of water extracted (ml)
$V_i$ = Volume of extract injected (µL)

12.2.5. Sediment/Soil/Sludge (on a dry weight basis) and Waste (normally on a wet weight basis)

<b>Concentration (µg/kg) = <math>\frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)(D)}</math></b>
where:
$A_x$ = Area of characteristic ion for compound being measured



$I_s$ = Amount of internal standard injected (ng).
$V_t$ = Volume of total extract, taking into account dilutions (i.e. for a 1 to 10 dilution of a 1 ml extract $V_t = 10,000 \mu\text{L}$ )
$A_{IS}$ = Area of characteristic ion for the internal standard
RRF = Relative response factor for compound being measured
$V_i$ = Volume of extract injected ( $\mu\text{L}$ )
$W_s$ = Weight of sample extracted or diluted in grams
D = % Dry weight of sample = 1.0 on an as received basis

### 13. Method Performance

13.1. The QA department measures method performance using a combination of annual method detection limit (MDL) studies, performance evaluation samples, and the monitoring of surrogate and spike recoveries.

13.1.1. Detection limits- detection limits for all analytes quantitated using this SOP are set using the low point of the initial calibration curve and validated via method detection limit studies.

13.1.2. MDL studies are performed each year for each analyte by each preparatory and analytical method (for example, analytes quantitated for in water samples will have MDL studies performed for both the liquid/liquid extraction and sep funnel extraction, and the associated mass spectrometry detection methods.)

13.1.3. MDL and reporting limit (RL) values may be found for each analyte in the ARI QAP

13.2. Laboratory precision and bias measurements are performed by monitoring surrogate and spike recoveries in samples and quality control samples.

13.3. Control limits are calculated by monitoring these recoveries. These control limits are disseminated to the bench chemists and LIMS administrator for use in monitoring method performance in real time. As these limits are updated regularly, their dynamic nature prevents their inclusion in this SOP. However, they may be found in the ARI QAP.

### 14. Pollution Prevention

14.1. All syringe rinsing must be performed over charcoal to minimize the exposure of the environment to solvent or extract.

14.2. All GC split vents will be connected to an exhaust vent.

14.3. All MS vacuum pumps will have a charcoal exhaust filter.

### 15. Data Assessment and Acceptance Criteria for QC Measure



- 15.1. Requirements relating to initial and continuing calibration are detailed in Section 10 of this document.
- 15.2. Method Blanks must be free of target analytes with a concentration greater than 1/2 their reporting limit.
- 15.3. Internal Standard EICP areas following the continuing calibration standard must meet the technical acceptance criteria listed in SOP Section 9.2.7.3.
- 15.4. Surrogate Recoveries for all sample, MB, LCS, MS, MD, SRM analyses must have acceptable surrogate recoveries based on ARI's current control charts.
- 15.4.1. These requirements do not apply to subsequent dilutions of samples where a prior analysis of the diluted sample extract shows acceptable surrogate recovery.
  - 15.4.2. Certain methods or clients may specify project specific surrogate recovery acceptance windows.
  - 15.4.3. When mandated by contract-specific requirements, corrective actions must be performed in response to failure to meet project specific surrogate acceptance criteria, even when the criteria are labeled as advisory in the reference method.
  - 15.4.4. Surrogate acceptance criteria are both matrix and concentration level specific (e.g. low level vs. medium level soils). When analyzing matrices or concentration levels for which no acceptance criteria are available, the closest approximation of available acceptance criteria may be provided as estimates for advisory purposes only.
- 15.5. Laboratory Control Samples (LCS)
- 15.5.1. The LCS recovery values should fall within the specified recovery acceptance limits. If an LCSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.
  - 15.5.2. LCS recovery acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Project or method specific limits may supersede laboratory acceptance criteria.
- 15.6. Matrix Spike/Matrix Spike Duplicates (MS/MSD)
- 15.6.1. Matrix Spike/Matrix Spike Duplicate recovery values should fall within the specified recovery acceptance limits. If a MSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.
  - 15.6.2. MS/MSD recovery and RPD acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Certain methods or clients may require project specific MS/MSD recovery and RPD acceptance windows.



## 15.7. Holding Times

15.7.1. Samples must be extracted within holding times (seven days for water samples and fourteen days for solid samples).

15.7.2. Extracts must be analyzed within the extract holding time (forty days from the initial date of extraction.)

15.7.3. In the event that re-extraction due to an out of control event requires that samples be re-extracted after their extraction holding time has elapsed (seven days for water and fourteen days for tissues/solids) the analyst should analyze and report both extraction sets, whenever practical, distinguishing between the initial extraction and re-extraction on all deliverables. This will document that the samples were originally extracted within holding times and may allow for comparisons that will determine whether any of the more volatile analytes were lost in the interval between extractions.

15.7.4. If any extracts are analyzed after the 40 day extract holding time has elapsed, the analyst shall document this in the analytical notes accompanying the data so that it may be included in the case narrative.

## 16. Corrective Actions for Out of Control of Unacceptable Data

### 16.1. Mass Spectrometer Tuning

16.1.1. When the MS does not produce an acceptable mass spectrum with 25 -50 µg/mL of DFTPP, re-inject the DFTPP. If the spectrum again fails to meet the criteria found in Appendix 20.3, the MS may need to be re-tuned.

16.1.2. If the re-tuned mass spectrum still fails to meet the criteria found in Appendix 20.3, the MS may need maintenance. The MS should be vented, and maintenance may include: replacing the filaments, cleaning the MS source, cleaning the MS quadrupoles, or replacing the electron multiplier.

16.2. Peak tailing factors- If the peak tailing factors exceed the limits found in Section 9.2.7.1.1.4, the chromatographic system may need maintenance. Inspect the system for leaks. Should none be found, perform maintenance on the chromatographic system. This maintenance includes, but is not limited to: replace the inlet liner and liner packing, clean the inlet liner, cleaning or replacing the inlet seal, cleaning or replacing the inlet body, replacing the split line, cleaning the split arm, clipping a length from the front of the column, or replacing the column. Any maintenance is to be documented in the "maintenance" section of the GC/MS run logbook for that instrument.

16.3. Initial Calibration/CCV failure- should RSD or %D failure be evident in either the initial calibration or the CCV, perform the maintenance found in Section 16.1.2. If the failure is deemed to be the result of an improperly prepared calibration standard, re-prepare the standard and



reanalyze it. Reanalyzing or replacing a single standard must NOT be confused with the practice of discarding individual calibration results for specific target compounds in order to pick and choose a set of results that will meet the RSD or correlation criteria for the linear model. The practice of discarding individual calibration results is addressed as a fourth alternative option, and is very specific as to how a set of results are chosen to be discarded. If a standard is reanalyzed, or a new standard is analyzed, then ALL of the results from the original analysis of the standard in question must be discarded. Further, the practice of running additional standards at other concentrations and then picking only those results that meet the calibration acceptance criteria is EXPRESSLY PROHIBITED, since the analyst has generated data that demonstrate that the linear model does not apply to all of the data.

#### 16.4. Internal Standards-

16.4.1. If the internal standards fail to meet their acceptance criteria, check calculations, the internal standard compound spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard response met their acceptance criteria.

16.4.2. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze the samples.

16.4.3. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will most likely involve instrument maintenance similar to the maintenance described in Section 16.1.2. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.4.4. If the above actions do not correct the problem, then the problem may be due to a sample "matrix effect". To determine if there is a matrix effect, take the following corrective action steps.

16.4.4.1. Reanalyze the sample extract. EXCEPTION: if the internal standard compounds responses and/or retention times in a sample used for a matrix spike and/or matrix spike duplicate were outside the acceptance criteria, then it should be reanalyzed only if the internal standard responses and/or retention times were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of unspiked duplicates of a given sample which is outside acceptance criteria.

16.4.4.2. If the corrective actions listed above do not prove matrix effect, then the problem with the initial analysis is deemed to be within the laboratory's control. Submit either both



sets of data or only the data from the reanalysis with the internal standard responses and retention times within acceptance limits.

16.4.4.3. If the corrective actions listed above prove matrix effect, then submit data from both analyses, distinguishing between the initial analysis and reanalysis on all deliverables.

16.4.4.4. If internal standard acceptance criteria are not met in an extract judged by the GC/MS analyst to be overly concentrated in background (e.g. hydrocarbons) and therefore damaging to the analytical system, the extract may be diluted prior to the reanalysis performed to prove matrix effect.

16.4.4.5. If a set of sample extracts from the same sample delivery group and/or samples collected from the same sample location are judged to be similar based on examination of the total ion chromatogram, it may be necessary only to reanalyze one extract or a subset of the extracts to prove matrix interference. Prior consultation with the Laboratory Manager, Project Manager and the client is necessary for this option to be used.

## 16.5. Surrogates

16.5.1. If the surrogate compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.5.2. If the surrogate recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the surrogate compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the surrogate compound recoveries meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of surrogate compound spiking solution was added either recalculate surrogate recoveries based on the actual amount of surrogate compound spiking solution added, or re-extract/reanalyze the sample, adding the correct amount of surrogate spiking solution. If the surrogate compound spiking solution and/or surrogate calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.5.3. If the surrogate recoveries were outside the lower recovery acceptance limit and the extract from the sample was cleaned up on a Gel Permeation Chromatograph (GPC) using an automated injection system, the laboratory shall verify that the proper amount was injected on the GPC column. If insufficient sample volume was injected on the GPC, the sample must be re-extracted and reanalyzed. Alternatively, the archived portion of the original extract must be subjected to GPC clean-up and reanalyzed.





16.5.4. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will mostly involve maintenance similar to the maintenance discussed in Section 16.1.2. Verify that the surrogate recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.5.5. If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was matrix effect, take the following corrective action steps.

16.5.5.1. Re-extract and reanalyze the sample. EXCEPTION: If surrogate compound recoveries in a sample used for a matrix spike and/or matrix spike duplicate were considered unacceptable, then it should be re-extracted/reanalyzed only if the surrogate compound recoveries were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of un-spiked duplicates of a given sample which is outside acceptance criteria.

16.5.5.2. If the surrogate compound recoveries meet acceptance criteria in the re-extracted/reanalyzed sample, then the problem with the initial analysis is deemed to be within the laboratory's control. Therefore, submit data only from the re-extraction/reanalysis if the re-extraction was performed within holding time, otherwise report both sets of data.

16.5.5.3. If the surrogate compound recoveries fail to meet the acceptance criteria in the re-extracted/reanalyzed sample, then submit data from both analyses, distinguishing between the initial analysis and the re-extraction/reanalysis on all deliverables.

16.6. Method Blanks- Corrective action for a method blank which fails acceptance criteria may involve re-extraction and reanalysis of all associated samples and/or "B" flagging of the associated sample data. Each occurrence will be evaluated on an individual basis upon consultation with the Project Manager, the client, the Laboratory Supervisor, and the Laboratory Manager.

#### 16.7. Laboratory Control Samples

16.7.1. If the LCS compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.7.2. If the LCS recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the LCS compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the LCS compound recoveries and RPDs (if applicable) meet



their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of LCS compound spiking solution was added either recalculate LCS recoveries based on the actual amount of LCS compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of LCS spiking solution. If the LCS compound spiking solution and/or LCS calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.7.3. If the LCS recoveries were outside the lower recovery acceptance limit and the extract from the sample was cleaned up on a Gel Permeation Chromatograph (GPC) using an automated injection system, the laboratory shall verify that the proper amount was injected on the GPC column. If insufficient sample volume was injected on the GPC, the samples must be re-extracted and reanalyzed. Alternatively, the archived portion of the original extract must be subjected to GPC clean-up and reanalyzed.

16.7.4. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the LCS recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.7.5. If the LCS compounds still fail to meet their acceptance criteria, re-extract and reanalyze the LCS and all associated samples and QC samples if deemed appropriate (i.e. after consideration of all batch QC data) or mandated by contract-specific requirements. Any decision to forgo re-extraction/reanalysis based on failure to meet LCS acceptance criteria will require approval of the Project Manager and the Lab Manager, at a minimum.

#### 16.8. Matrix Spike/Matrix Spike Duplicates

16.8.1. If the MS/MSD compounds fail to meet their advisory acceptance criteria, it may be necessary to reanalyze the extracts

16.8.2. If this fails to bring the recoveries or RPDs into compliance, check calculations, sample preparation logs, the MS/MSD compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the MS/MSD compound recoveries and RPD's (if applicable) meet their advisory acceptance criteria. If the sample preparation logs indicate that the incorrect amount of MS/MSD compound spiking solution was added either recalculate MS/MSD recoveries based on the actual amount of MS/MSD compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of MS/MSD spiking solution. If the MS/MSD compound spiking solution and/or MS/MSD calibration solution was improperly



prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

- 16.8.3. If the MS/MSD recoveries were outside the lower advisory recovery acceptance limit and the extract from the sample was cleaned up on a Gel Permeation Chromatograph (GPC) using an automated injection system, the laboratory shall verify that the proper amount was injected on the GPC column. If insufficient sample volume was injected on the GPC, the samples must be re-extracted and reanalyzed. Alternatively, the archived portion of the original extract must be subjected to GPC clean-up and reanalyzed.
- 16.8.4. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the MS/MSD recoveries meet their advisory acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.
- 16.8.5. If the MS/MSD compounds still fail to meet their advisory acceptance criteria, an assessment of batch QC data is necessary in order to determine the possibility of a matrix effect. If the RPDs are within acceptance limits (particularly for non-native MS/MSD analytes) and all of the batch QC elements which are not affected by the sample matrix are in control (e.g. method blank, LCS, calibration checks) and there is no evidence of improper spiking levels, the poor recovery may be attributed to matrix effects. Additional consideration also needs to be given to the ratio of spike to native analyte, as well as sample heterogeneity, as these factors may have an adverse effect on both spike recovery and RPD values. If matrix effect is determined to be the cause, the associated data must be flagged, but re-preparation and reanalysis is not required. Examination of spike recoveries in the matrix spike duplicate is also pertinent to assessing matrix effects.
- 16.8.6. If any of the batch QA elements which are not affected by the sample matrix are out of control, or there is evidence that spiking may have been improperly performed, the matrix spike and matrix spike duplicate must be re-extracted and reanalyzed. If insufficient sample is available or if the extraction holding time has elapsed, the laboratory must notify the client and flag the associated data and/or document the problem in the narrative.
- 16.9. Sample Dilution- When the response (EICP) for any analyte exceeds the highest response in the working range of the calibration, the analyte response must be qualified with an "E" flag, the sample diluted to reduce the analyte's response into the calibration range and the diluted sample re-analyzed.
- 16.9.1. Additional internal standard must be added to the diluted extract to maintain the required 2 ng/μL of each internal standard in the extracted volume.



16.9.2. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

16.9.3. When ions from a target compound in the sample saturate the detector the analyst must:

16.9.3.1. Flag all affected analytes with an S flag.

16.9.3.2. Analyze an Instrument Blank consisting of clean solvent until the system has been decontaminated. It is advisable to reanalyze the sample run directly after the sample with the saturated analyte and compare the original and reanalysis values to look for carryover.

## **17. Contingencies for Handling Out-of-Control or Unacceptable Data**

17.1. See Section 16.1 for guidance on dealing with out-of-control tuning events.

17.2. See Sections 16.2 and 16.3 for guidance on dealing with out-of-control events related to initial calibrations, peak tailing factors, or DDT breakdown.

17.3. See Section 16.4 for guidance on dealing with internal standard out-of-control events.

17.4. See Section 16.5 for guidance on dealing with surrogate out-of-control events.

17.5. See Section 16.6 for guidance on dealing with method blank related out-of-control events.

17.6. See Section 16.7 for guidance on dealing with laboratory control sample related out-of-control events.

17.7. See Section 16.8 for guidance on dealing with MS/MSD related out-of-control events.

## **18. Waste Management**

18.1. All extract vials must be disposed of by placing them in the blue hazardous waste drum in the station set aside for this purpose (Satellite Accumulation Station GC-2.) No vials may be thrown in the trash or receptacles not expressly designated for this purpose.

18.2. All solvents must be disposed of by pouring them out over charcoal. No solvent may be poured down the drain or disposed of in any other non-hygienic manner.

18.3. All spent charcoal must be disposed of by placing it in the charcoal disposal bin located in the extractions lab.

## **19. Method References**

19.1. "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8270D, Test Methods for Evaluating Solid Waste (SW-846), Revision 4, January, 1998.

19.2. USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration Revision OLM03.1, August, 1994.



19.3. "Determinative Chromatographic Separations": Method 8000C, Test Methods For Evaluating Solid Waste (SW-846), Revision 3, March, 2003.

19.4. Department of Defense QSM, Version 3, Revision 5, June, 2003.

## **20. Appendices**

20.1. Appendix 20.1: Manual Data Integration

20.2. Appendix 20.2: GC-MS-SIM – Organophosphorous Pesticide Target Analytes, Scan Descriptors, Quantitation Ions

20.3. Appendix 20.3: DFTPP key ions and ion abundance criteria.

20.4. Appendix 20.4: GC-MS Conditions

20.5. Appendix 20.5: GC and GC/MS Semivolatile Organics Logbook (Example)

20.6. Appendix 20.6: Peak Tailing Factor Calculation

20.7. Appendix 20.7: Extract Screening Procedure



## Appendix 20.1

### Manual Integration

**Manual Adjustment:** Modern chromatographic instruments include computer software to identify a detector response as a chromatographic peak, characterize that peak and determine the relative height or area of the signal. The software utilizes parameters (threshold, slope, etc.) that are adjusted by the instrument operator to optimize the results. A single set of operator controlled settings that determine peak characteristics for an entire data file (or sets of data files) is defined as an “automated procedure.” An automated procedure often characterizes chromatographic peaks incorrectly. ARI requires that trained analysts identify and resolve these errors using an alternative automated procedure or a “manual adjustment” of the data. Manual adjustment is defined as the process used by an analyst to adjust an individual peak or a subset of data in a chromatographic file.

The settings for the routine automated procedure normally used to process chromatographic data for this SOP are listed below for both the total (or reconstructed) ion chromatogram (RIC) used for the quantitation of TIC's and for the extracted ion current profile (EICP) used for the quantitation of target compound analytes and surrogates.

<b>Parameter</b>	<b>RIC</b>	<b>EICP</b>
Peak Detection - Start	0.200	0.200
Peak Detection - End	0.000	0.000
Bunch	1	1
Peak Smoothing	on	off
Area Cutoff	0 %	5 %
Maximum Peaks to Detect	200	100
Baseline Reset	3 - 5	5
Set Valley	100	100

Trained analysts may substitute one automated procedure for another in order to optimize peak characteristics. The use of an alternate automated procedure must be permanently documented using either a software generated log file or analyst notes.

Manual adjustment of chromatographic peak characteristics will be used to correct the results of an automated procedure that, in the trained analyst's opinion, are clearly incorrect and will result in erroneous peak identification, integration or quantification.



Manual adjustment will be implemented in a reasonable and consistent manner. The adjustment shall include only the area attributed to the specific target compound. The area adjusted shall not include baseline background noise. The area adjusted shall not extend past the points where the sides of the peak intersect with the baseline noise.

All manually adjusted data shall be clearly identified for approval in the data review process. A permanent record of all manual adjustments shall be maintained in both electronic and hardcopy versions of the data.

Manual adjustment of chromatographic data will not be used to falsify data for any purpose or as a substitute for corrective action on the chromatographic system. Falsification of data through the use of manual peak adjustment is unethical, unlawful and will result in the termination of the offending analyst.

An analyst who changes an integration must identify the manual integration on the quantitation report then initial and date the report.

All manually integrated reported data will be flagged, and, when mandated by project-specific requirements, the final data report and associated documentation must provide justification for manual integrations.



**Appendix 20.2**  
**GC-MS-SIM – Organophosphorous Pesticide Target Analytes,**  
**Scan Descriptors, Quantitation Ions**

Scan Group	Start Time (min)	Ions Scanned	Internal Standard Associated Analyte	CAS Number	Primary Ion	RL Water µg/L <sup>3</sup>	RL Soil µg/Kg <sup>4</sup>
1	0.00	79, 108, 109, 136, 137, 185	Naphthalene d8 – IS <sup>1</sup>		136		
			Dichlorvos	62-73-7	109	0.08	8
2	8.00	99, 109, 127, 160, 161, 162, 164, 192	Mevinphos (Phosdrin)	7786-34-7	127	0.04	4
			Acenaphthene-d10 – IS <sup>1</sup>		164		
3	10.30	60, 88, 89, 97, 99, 126, 155, 158, 211	Demeton-S	8065-48-3	88	0.20	20
			Ethoprop (Ethoprofos)	13194-48-4	158	0.20	20
			Tributyl phosphate – SS <sup>2</sup>		99		
4	10.90	75, 79, 93, 97, 109, 121, 127, 145, 192, 322	Dicrotophos	141-66-2	127	0.20	20
			Monocrotophos	6923-22-4	127	0.50	50
			Sulfotepp	3689-24-5	322	0.20	20
			Phorate	298-02-2	75	0.04	40
5	11.40	60, 87, 88, 93, 125	Dimethoate	60-51-5	87	0.20	20
6	11.80	88, 89, 97, 137, 179, 187, 188, 199	Phenanthrene-d10 – IS <sup>1</sup>		188		
			Diazinon	333-41-5	137	0.04	40
			Disulfoton	298-04-4	88	0.04	40
7	12.70	109, 125, 146, 160, 179, 188, 263, 285, 287, 127,	Methyl Parathion	298-00-0	109	0.20	20
			Fenchlorophos (Ronnel)	299-84-3	285	0.20	20
			Malathion	121-75-5	173	0.20	20
8	13.71	97, 105, 109, 125, 127,	Fenthion	55-38-9	278	0.20	20
			Chlorpyrophos	2921-88-2	197	0.04	4





**Appendix 20.2**  
**GC-MS-SIM – Organophosphorous Pesticide Target Analytes,**  
**Scan Descriptors, Quantitation Ions**

Scan Group	Start Time (min)	Ions Scanned	Internal Standard Associated Analyte	CAS Number	Primary Ion	RL Water $\mu\text{g}/\text{L}^3$	RL Soil $\mu\text{g}/\text{Kg}^4$
		139, 153, 193, 197, 209, 267, 269, 278, 291, 297, 298, 314, 323	Parathion	56-38-2	291	0.20	20
			Trichloronate	327-98-0	109	0.04	4
			Chlorfenvinphos	470-90-6	267	0.20	20
			Crotoxyphos	7700-17-6	105	0.20	20
9	14.75	109, 113, 147, 162, 169, 170, 267, 329	Tetrachlorvinphos (Stirophos)	22248-79-9	109	0.20	20
			Tokuthion	3464-346-4	162	0.20	20
			Merphos Oxone	150-50-5	169	0.50	50
10	15.60	97, 125, 139, 140, 153, 156, 231, 293, 325, 326,	Fensulfothion	115-90-2	293	0.50	50
			Ethion	563-12-2	231	0.20	20
			Bolstar (Sulprophos)	35400-43-2	156	0.20	20
			Triphenyl Phosphate – SS <sup>2</sup>		326		
11	17.00	141, 157, 169, 236, 240	EPN	2104-64-5	157	0.20	20
			Chrysene-d12 – IS <sup>1</sup>		240		
12	17.60	77, 97, 109, 132, 160, 226	Guthion	86-50-0	160	0.50	50
			Coumaphos	56-72-4	109	0.20	20
			Alachlor	15972-60-8	160	0.20	20

1 – Compounds followed by (IS) are internal standards

2 – Compounds followed by (SS) are surrogate standards

3 – Reporting Limit based on 500 mL sample and a 0.5 mL final volume

4 – Reporting Limit based on 10 g sample and a 1.0 mL final volume



## Appendix: 20.3 DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

<b>Mass</b>	<b>Ion Abundance Criteria</b>
51	30-80 % of mass 198
68	< 2 % of mass 69
70	< 2 % of mass 69
127	25-75 % of mass 198
197	< 1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	> 0.75 of mass 198
441	Present but less than mass 443
442	40-110 % of mass 198
443	15-24 % of mass 442



## Appendix 20.4 GC-MS Conditions

Quantitation Mass range:	12 Selected Mass Ranges
Tuning Mass Range	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	60°C, hold for 2 minutes
Temperature program:	60-130°C at 30C°/min 130-330°C at 20C°/min
Final temperature:	330°C, hold for 5 minutes
Injector temperature:	250°C
Transfer line temperature:	300°C
Source temperature:	According to manufacturer's specifications (170-180°C for 5972 MSD, 230°C for 5973)
Injector:	Grob-type, splitless/split
Sample volume:	1-2 µL
Carrier gas:	Helium at 30 cm/sec.



## Appendix 5 GC and GC/MS Semivolatile Organics Logbook (Example)

**Analytical Resources Inc.: Organics Instrument Log**  
NT-2 Serial No.: 82321977

Date: \_\_\_\_\_ Analysis: \_\_\_\_\_ Analyst: \_\_\_\_\_  
 GC Program: \_\_\_\_\_ Column No: \_\_\_\_\_ Column Type: \_\_\_\_\_  
 Instrument Tune (.U or .CT.): \_\_\_\_\_ EM Voltage: \_\_\_\_\_  
 Calibration File: \_\_\_\_\_ Curve Date: \_\_\_\_\_

IS/SS	Ical/Ccal	LCS/ICV

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**Maintenance / Comments**

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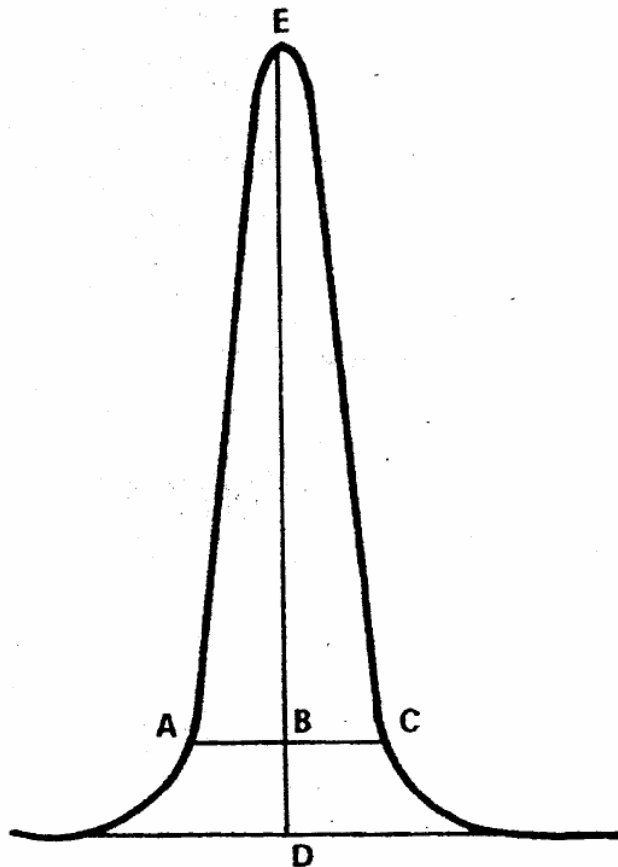
---

**Maintenance Verification** (Identify ICal or CCal that demonstrates the instrument is in control):  
 Every line must contain information or be lined out. Make all entries legible. Start a new page for each QC period.



## Appendix 6 Peak Tailing Factor Calculations

$$RF = \frac{(A_p)(C_p)}{(A_s)(C_s)}$$



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

**Example calculation:** Peak Height = DE = 100 mm  
10% Peak Height = BD = 10 mm  
Peak Width at 10% Peak Height = AC = 23 mm  
AB = 11 mm  
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$



## Appendix 7 Extract Screening Procedure

Spike a 0.3 ml aliquot of the 0.5-1ml extract obtained from sample preparation with 6  $\mu\text{L}$  of the internal standard solution just prior to analysis. This is the equivalent internal standard concentration of 2  $\text{ng}/\mu\text{L}$  of each standard in the sample. This aliquot should be prepared in an amber glass autosampler vial and sealed with a Teflon lined crimp cap.

Analyze the 0.3 ml aliquot by GC/MS. The injection volume must be the same volume used for the calibration standards. The recommended GC/MS operating conditions to be used are specified in Section 11.6.

If the response for any target analyte exceeds 10/50  $\text{ng}/\mu\text{l}$  (high point of the initial calibration curve for PNAs or PCP respectively), extract dilution must take place. See Section 16 for guidance on dilutions.



**Analytical Resources, Incorporated**  
Analytical Chemists and Consultants

## **Standard Operating Procedure**

### **Separatory Funnel Extraction Aqueous Samples for Herbicide Analysis**

**SOP 324S  
Version 012**

**Revision Date: 7/18/06  
Effective Date: 7/18/06**

Prepared By:

Mark Weidner

Approvals:

Jim N. Jelic  
Laboratory / Section Manager

David R. Mitchell  
Quality Assurance







## 1. Scope and Application

- 1.1. This method details the separatory funnel extraction procedure for water samples or TCLP extracts in preparation for 8151A, chlorinated herbicide analysis. This SOP is written to meet the requirements of SW-846, Method 3510-C.

## 2. Definitions

- 2.1. ECD (Electron Capture Detector) – A gas chromatographic detector that is specific and sensitive to halogenated organic compounds.
- 2.2. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.
- 2.3. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the sample matrix on a method's recovery efficiency.
- 2.4. Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 2.5. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 2.6. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to evaluate extraction efficiency..

## 3. Interferences

- 3.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.
- 3.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S.
- 3.3. Some lots of methylene chloride may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have is verified free of contaminants
- 3.4. Laboratory supplies and equipment are potential sources of interfering contamination
  - 3.4.1. Make sure items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.



- 3.4.2. Use glassware that is thoroughly cleaned, kiln fired (SOP # 301S) and solvent rinsed.
- 3.5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.
- 3.6. Naturally occurring non-petroleum organic material may contribute to biogenic interferences. Acid/silica gel clean-up can be used to assist in the reduction or elimination of these interferences.

#### **4. Safety**

- 4.1. The toxicity or carcinogenicity of each reagent used in this SOP is not been precisely defined. Treat each chemical compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.
- 4.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection
- 4.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT).
- 4.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted as the need may arise. The MSDS file is located in the central project management area. MSDS are also available online, at <http://hazard.com/MSDS/>.
- 4.5. Environmental Samples may contain hazardous waste; treat them as potential health hazards.
- 4.6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be stored for repair.
- 4.7. Use nitrile (blue) gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride.
- 4.8. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware. Do not glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be set aside for repair.

#### **5. Equipment and Supplies**

- 5.1. Equipment
  - 5.1.1. Personal protective equipment (PPE)
    - 5.1.1.1. Gloves
    - 5.1.1.2. Laboratory coat
    - 5.1.1.3. Goggles



- 5.1.2. wide range pH paper
- 5.1.3. Glass etcher / engraver
- 5.1.4. Water Bath, Precision Scientific 51220042 or equivalent
- 5.1.5. Zymark TurboVap LV (Set at 40°C)
- 5.1.6. Centrifuge Beckmann GP or equivalent
- 5.1.7. Hamilton gastight syringes: 100µL, 250µL, 10ml
- 5.2. Glassware
  - 5.2.1. Graduated cylinder, glass, pre-cleaned graduated 'to deliver'. One 100, 1000 or 2000 mL cylinder per sample depending on volume of sample to be extracted
  - 5.2.2. Separatory funnel (SF), glass with PTFE stopper and stopcock plug (one for each sample)
    - 5.2.2.1. 1000 mL SF for 500 mL samples
    - 5.2.2.2. 250mL SF for 100 mL (TCLP) samples
  - 5.2.3. Erlenmeyer flask, Glass, 250 mL (one for each sample)
  - 5.2.4. Erlenmeyer flask, glass, 500 mL
  - 5.2.5. Centrifuge bottles, glass, 200 mL
  - 5.2.6. Powder Funnel, glass, 70mm
  - 5.2.7. Kuderna-Danish concentrating apparatus, glass, single walled with 10mL concentrator tube, 500mL concentrator flask and three-ball Snyder Column
  - 5.2.8. Turbo Tube, glass, 60 mL with 0.5 mL reservoir
  - 5.2.9. 'Tilt-a-pet' dispenser, glass, 30 or 60 mL used to add a measured volume of extracting solvent to the sample in a separatory funnel
  - 5.2.10. Pasteur Pipette, borosilicate glass, 5 ¼ inch, disposable
  - 5.2.11. Centrifuge tube, borosilicate glass, 15mL.
- 5.3. Supplies
  - 5.3.1. Boiling Chips – PTFE (Chemware)
  - 5.3.2. Wide Sharpie Permanent Pen
  - 5.3.3. Pink label tape
  - 5.3.4. Kimwipe

## 6. Reagents and Standards

- 6.1. Hexane, residue analysis grade.
- 6.2. Methylene Chloride, residue analysis grade.
- 6.3. Acetone, residue analysis grade.
- 6.4. 7:3 methylene chloride/hexane mixture.



- 6.5. 7:3 methylene chloride/acetone mixture.
- 6.6. 1 to1 sulfuric acid solution is prepared by adding 500 mL of sulfuric acid to 500 mL of organic free water.
- 6.7. 10N sodium hydroxide is prepared by adding 400g of sodium hydroxide pellets to 1L of organic free water
- 6.8. Anhydrous acidified sodium sulfate is prepared by baking in kiln at 400 for 4 hours. To 800 grams of anhydrous sodium sulfate add 200ml of diethyl ether containing 1 ml of concentrated sulfuric acid. Mix the sodium sulfate well and evaporate the diethyl ether under vacuum.
- 6.9. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.
- 6.10. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (form 10076F)

## **7. Sample Collection, Preservation, Shipment and Storage**

- 7.1. Store all water samples at 2°C to 6°C. Warm the samples to ambient temperature prior to extraction.
- 7.2. Required holding time for all water samples is 7 days from sample collection. Extract aqueous samples within seven days of collection. When holding times are or may be compromised inform your supervisor immediately
- 7.3. TCLP and SPLP extracts are considered water samples with a 7 holding time.
- 7.4. Some samples are shared with the metals and or conventionals laboratories. These samples are placed in a share bin in Refrigerator 5. SOP 1019S includes procedures for handling shared samples.

## **8. Quality Control**

- 8.1. One method blank will be extracted for each batch of 20 or fewer samples.
- 8.2. One LCS (spike blank) will be extracted for each batch of 20 or fewer samples.
- 8.3. For TCLP, one matrix spike will be extracted for each batch of 20 or fewer samples.
- 8.4. Upon client's request one MS/MSD will be extracted for each batch of 20 or fewer samples, providing sufficient sample is available for the analysis. If insufficient volume is received for MS/MSD for each batch of 20 samples, a LCS Duplicate will be extracted.
- 8.5. To verify that surrogate and matrix spiking is accurate, spiking will be witnessed and documented by a second laboratory technician.
- 8.6. For the procedure, take things slowly and carefully and write down any noted problems or deviations on the Analyst Notes Form (Form 3056F). Even if you believe the deviation will not



affect the final result, you may not be aware of how dramatically some of these changes may affect surrogate and matrix spike recoveries. If you run into technical problems that cannot be answered by your supervisor, notify the GC supervisor or the senior chemist immediately for instructions.

- 8.7. Use a permanent marker to label all glassware and vials.
- 8.8. Always check the sample ID when transferring or vialing samples and extracts, or when changing glassware.
- 8.9. Constant attention must be given to extracts when they are on the water bath or turboVap. Check extracts often when solvent levels are low to ensure extracts do not concentrate to dryness.
- 8.10. Review the Special Analytical Requirements Form (Form 3002F) prior to extracting samples to determine how the samples are to be extracted.
- 8.11. The LIMS Data entry person will review all Extraction Requirement sheets (Form 3002F) and bench sheets before distribution.
- 8.12. All logbooks are reviewed monthly for completeness and accuracy by the laboratory supervisor
- 8.13. The QA section will periodically review the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

## 9. Calibration and Standardization

- 9.1. Not Applicable

## 10. Procedure

- 10.1. Review the special analytical requirements form prior to extracting samples to determine if special procedures are required.
- 10.2. Fill out a bench sheet for the job.
- 10.3. Warm the samples to room temperature.
- 10.4. Label each separatory funnel with a piece of pink tape containing the following information: ARI job number, sample ID letter, matrix ID, type of extraction (Herb, or Herb TCLP) and for the Method Blank (MB) and the LCS, the date.
- 10.5. Measure 500 mL deionized water into each of two 1000 mL separatory funnels, one to use as a MB and one to use as the LCS. For TCLP extraction, prepare two 250 mL separatory funnels with 100 mL of TCLP solution (i.e. the MB is extracted along with the sample by the Metals Prep Laboratory), one for use as a MB and one for use as the LCS.
- 10.6. Samples received in bottles for the correct volume of 500 mL are marked with a glass etcher at the meniscus line. Transfer the sample to the Separatory Funnel. The bottles are rinsed with



- 30 mL of DI water containing one pipette of 10N sodium hydroxide. The rinsate is added to the separatory funnel and the contents are mixed well. Verify that the pH of the sample is  $> 12$  using a pipette and broad range pH paper. Add additional 10N sodium hydroxide drop wise as needed until  $\text{pH} \geq 12$ .
- 10.7. Sample volumes are determined by filling the bottles to the etched marks with tap water and measuring with a graduated cylinder calibrated to contain. Samples received in incorrect bottles for volumes (i.e. 1 L bottles for a 500 mL analysis) will be measured in a graduated cylinder, using one cylinder for each sample. If there are insufficient cylinders for the batch, the cylinder will be cleaned before it is used for another sample (See ARI SOP 301S, Contrex AP Bath, Acid Bath, D.I. Water Bath, Acetone Rinse, Methylene Chloride Rinse) (NOTE: no residual acetone should be left from the acetone rinse).
  - 10.8. Add surrogates to each sample, MB, LCS and MS as specified on the extraction requirement form, 3002F. In addition to the surrogates, the LCS and MS receive a full list herbicide spike. To verify that each surrogate and spike addition is accurate, each will be witnessed and documented by another laboratory technician.
  - 10.9. The samples are mixed well and allowed to stand at room temperature for one hour to allow for the hydrolysis of herbicide esters to the free acids.
  - 10.10. After one hour add 60 ml of methylene chloride to the samples and shake the separatory funnel vigorously for two minutes. Allow the phases to separate and drain off the bottom layer, (methylene chloride), to a waste beaker.
  - 10.11. Acidify the sample to  $\text{pH} < 2$  with 1:1 sulfuric acid solution. Mix well. Use broad-range pH paper to check the pH.
  - 10.12. Prepare a 250 ml Erlenmeyer flask for each sample by adding approximately 10 gm of acidified sodium sulfate.
  - 10.13. Add 60ml of 7:3 methylene chloride/acetone to the separatory funnel and shake for two minutes, venting frequently. Drain the bottom layer, (methylene chloride), into the corresponding Erlenmeyer flask. Extract the sample two more times using 60 ml of 7:3 methylene chloride/hexane. Each time the bottom layer should be drained into the corresponding Erlenmeyer flask.
  - 10.14. Discard the water in the separatory funnel and transfer the sample label to the 250 ml Erlenmeyer containing the extract. Additional anhydrous acid sodium sulfate may be added to the extract in sufficient quantity so that some sodium sulfate remains "free flowing". Allow the extract to stand for one hour to remove any water in the solvent.



- 10.15. Assemble a Kuderna-Danish (K-D) concentrator by attaching a methylene chloride rinsed 10 mL concentrator tube to a methylene chloride rinsed 500 mL evaporation flask. Add 2-3 clean boiling chips to the evaporation flask. A drying column is not needed.
- 10.16. Verify that the anhydrous sodium sulfate is “free flowing” in each extract. If it is not, add more anhydrous acidified sodium sulfate and wait 10-15 minutes before proceeding. Pour the extract into the 500 mL KD evaporator flask, being careful not to pour any sodium sulfate into the flask. Rinse the Erlenmeyer flask once with 40ml of hexane and add this to the KD as well. Transfer the label with the sample ID to the K-D. Repeat for each sample.
- 10.17. Discard the anhydrous sodium sulfate in the waste bucket marked for halogenated waste. Attach a methylene chloride rinsed three-ball Snyder column, and put the entire apparatus on a water bath set at approximately 100° C. Wet the inside of the Snyder column with 1-2 mL of hexane before boiling starts.
- 10.18. When the extract has concentrated to 4-8 mL, remove the apparatus from the water bath. Place the apparatus in a cooling rack and allow cooling for about ten minutes.
- 10.19. Remove the Snyder column and dry all water from the joint between the flask and the concentrator tube with a Kimwipe. Disassemble the tube and flask, and transfer the label with the sample ID to the tube.
- 10.20. Use a Pasteur pipete to transfer the extract into a 15 mL disposable centrifuge tube equipped with a Teflon lined phenolic screw cap. Adjust the volume to 10.0 ml with hexane and transfer the label with the sample ID and deliver samples to the GC lab for esterification and chromatographic analysis.

## 11. Data Analysis and Calculations

- 11.1. Not Applicable

## 12. Method Performance

- 12.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.
- 12.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

## 13. Pollution Prevention

- 13.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are



- 13.2. Pour any residual wastewater into the container in the Water Extraction Lab fume hood.
- 13.3. Do not discard organic solvent into the sink. Pour all waste solvent into the labeled 55-gallon drum located in the Hazardous Waste Storage Area.
- 13.4. Disposed expired standards into the designated barrel in the hazardous waste room.
- 13.5. Samples that are designated as hazardous waste by the LIMS “Hazardous Report” must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

#### **14. Data Assessment and Acceptance Criteria for Quality Control Measures**

- 14.1. Not Applicable

#### **15. Corrective Actions for Out of Control Events**

- 15.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.
- 15.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Section 22.2.

#### **16. Contingencies for Handling Out-of-Control or Unacceptable Data**

- 16.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a ‘Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

#### **17. Waste Management**

- 17.1. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled “Solvent Contaminated Solids” located in the Hazardous Waste Room when the accumulation pails are full.
- 17.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled “Chlorinated Solvents” located in the Hazardous Waste Storage Area
- 17.3. Disposed expired standards into the designated barrel in the hazardous waste room.
- 17.4. Place samples that designate as hazardous using the LIMS “Hazardous Report” in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions





must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.

17.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.

17.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

## **18. Method References**

18.1. U.S. EPA, "Separatory Funnel Liquid-Liquid Extraction", (SW-846), Method 3510C, Rev. 3, December, 1996.

18.2. U.S. EPA, "Acid-Base Partition Cleanup", (SW-846), Method 3650, Rev. 1, July, 1992.

18.3. (SW-846) "Chlorinated Herbicides by GC Using Methylation or Pentafluorbenzylation Derivatization", Method 8151A, Rev.1, December, 1996

# **Standard Operating Procedure**

## **Extraction of Soil or Sediment Samples For Herbicide Analysis (EPA Method 8151A)**

**SOP 325S  
Version 010**

**Revision Date: 02/23/06  
Effective Date: 02/23/06**

Prepared by:

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Approvals:

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Laboratory/Section Manager

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Quality Assurance





## 1. Scope and Application

- 1.1. This method details the extraction procedure for soil samples by to meet the requirements of SW-846, Method 8151, and Method 3550B.

## 2. Summary of the Procedure

- 2.1. Sediment samples are acidified and extracted with sonication into a suitable solvent. Because samples may contain herbicides as both free acids and esters, the extracts are subjected to a hydrolysis step that converts the esters into the acid form.
- 2.2. Prior to analysis the herbicides are converted into the methyl esters by derivatization with diazomethane in the gas chromatography laboratory.

## 3. Definitions

- 3.1. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.
- 3.2. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the sample matrix on a method's recovery efficiency.
- 3.3. Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 3.4. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 3.5. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

## 4. Interferences

- 4.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.



- 4.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.
- 4.3. Some lots of methylene chloride may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have is verified free of contaminates
- 4.4. Laboratory supplies and equipment are potential sources of interfering contamination
  - 4.4.1. Make sure items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.
  - 4.4.2. Use glassware that is thoroughly cleaned, kiln fired (SOP # 301S) and solvent rinsed.
- 4.5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.

## 5. Safety

- 5.1. The toxicity or carcinogenicity of each reagent used in this SOP is not been precisely defined. Treat each chemical compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.
- 5.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection
- 5.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT).
- 5.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted as the need may arise. The MSDS file is located in the central project management area. MSDS are also available online, at <http://hazard.com/MSDS/>.
- 5.5. Environmental Samples may contain hazardous waste; treat them as potential health hazards.
- 5.6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be stored for repair.
- 5.7. Use nitrile (blue) gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride.
- 5.8. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware. Do not glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be set aside for repair.

## 6. Equipment and Supplies



## 6.1. Equipment

### 6.1.1. Personal protective equipment (PPE)

6.1.1.1. Gloves

6.1.1.2. Laboratory coat

6.1.1.3. Goggles

6.1.2. Water Bath, Precision Scientific 51220042 or equivalent

6.1.3. Zymark TurboVap LV (Set at 40°C)

6.1.4. UltraSonic Mixer – Branson 450 with ¾" titanium horn

6.1.5. Drying oven (VWR BIOE) set at 105 C (±5)

6.1.6. Analytical Balance- Top loading, readable to 0.02g

6.1.7. Centrifuge Beckmann GP or equivalent

6.1.8. Hamilton gastight syringes: 100µL

6.1.9. Gas Chromatograph: Analytical system complete with gas chromatograph, and all required accessories including detectors, analytical columns, recorder, gases, and syringes. A data system for measuring peak heights or peak areas is also required.

6.1.9.1. Detectors:

6.1.9.2. Electron capture detector (ECD)

6.1.9.3. Flame ionization detector (FID)

6.1.9.4. Column: DB-5M (0.53 mm ID)

6.1.9.5. Recorders: HP 4 + (Printer)

## 6.2. Glassware

6.2.1. Erlenmeyer flask, Glass, 250 mL (one for each sample)

6.2.2. Centrifuge bottles, glass, 200 mL

6.2.3. Beaker, glass, 400mL (one for each sample)

6.2.4. Powder Funnel, glass, 100 mm

6.2.5. Kuderna-Danish concentrating apparatus, glass, single walled with 10mL concentrator tube, 500mL concentrator flask and three-ball Snyder Column

6.2.6. Turbo Tube, glass, 60 mL with 0.5 mL reservoir

6.2.7. 'Tilt-a-pet' dispenser, glass, 30 or 60 mL used to add a measured volume of extracting solvent to the sample in a separatory funnel

6.2.8. Drying column, glass, micro (16mm x 297mm)

6.2.9. Vial auto sampler clear glass, 2.5mL with PTFE lined crimp cap.

6.2.10. Pasteur Pipette, borosilicate glass, 5 ¼ inch, disposable

6.2.11. Glass stir rod



### 6.3. Supplies

- 6.3.1. Spatula, stainless steel
- 6.3.2. Weighing dish, Aluminum
- 6.3.3. Filter Medium, 0.45 $\mu$ m, Whatman Puridisc® (13mm PTFE) or equivalent
- 6.3.4. Filter Medium, 0.45 $\mu$ m, Whatman Puridisc® (25mm PTFE) or equivalent
- 6.3.5. Filter Medium, 0.45 $\mu$ m Millex filter (13mm or 25mm PTFE)
- 6.3.6. Filter Medium 0.45  $\mu$ m Millipore filter (25 mm PTFE)
- 6.3.7. Boiling Chips – PTFE (Chemware)
- 6.3.8. Glass wool, acidified, prepared by heating at 400°C for 4 hours. Followed by soaking in 0.05% HCL in Acetone (0.05mL conc. HCl in 1.0 L Acetone) then air drying.
- 6.3.9. Wide Sharpie Permanent Pen
- 6.3.10. Pink label tape
- 6.3.11. Kim wipe

### 7. Reagents and Standards

- 7.1. Acetone, high purity
- 7.2. Hexane, high purity
- 7.3. Methylene Chloride, high purity
- 7.4. Concentrated sulfuric acid
- 7.5. A 7:3 methylene/hexane mixture
- 7.6. A 7:3 methylene/acetone mixture
- 7.7. A 37% Potassium hydroxide (KOH) solution
- 7.8. 1:1 sulfuric acid solution is prepared by adding 500mL of sulfuric acid to 500mL of organic free water
- 7.9. Glass wool, acidified, prepared by heating at 400°C for 4 hours. Followed by soaking in 0.05% HCL in diethyl ether (0.05mL conc. HCl in 1.0 L diethyl ether) then air drying.
- 7.10. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.
- 7.11. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (form 10076F)

### 8. Sample Collection, Preservation, Shipment and Storage

- 8.1. Store all water samples at 2°C to 6°C. Warm the samples to ambient temperature prior to extraction.



- 8.2. Required holding time for all water samples is 7 days from sample collection. Extract aqueous samples within seven days of collection. When holding times are or may be compromised inform your supervisor immediately
- 8.3. TCLP and SPLP extracts are considered water samples with a 7 holding time.
- 8.4. Some samples are shared with the metals and or conventionals laboratories. These samples are placed in a share bin in Refrigerator 5. SOP 1019S includes procedures for handling shared samples.

## 9. Quality Control

- 9.1. One method blank will be extracted for each batch of 20 or fewer samples.
- 9.2. One LCS (spike blank) will be extracted for each batch of 20 or fewer samples.
- 9.3. For TCLP, one matrix spike will be extracted for each batch of 20 or fewer samples.
- 9.4. Upon client's request one MS/MSD will be extracted for each batch of 20 or fewer samples, providing sufficient sample is available for the analysis. If insufficient volume is received for MS/MSD for each batch of 20 samples, a LCS Duplicate will be extracted.
- 9.5. To verify that surrogate and matrix spiking is accurate, spiking will be witnessed and documented by a second laboratory technician.
- 9.6. For the procedure, take things slowly and carefully and write down any noted problems or deviations on the Analyst Notes Form (Form 3056F). Even if you believe the deviation will not affect the final result, you may not be aware of how dramatically some of these changes may affect surrogate and matrix spike recoveries. If you run into technical problems that cannot be answered by your supervisor, notify the GC supervisor or the senior chemist immediately for instructions.
- 9.7. Use a permanent marker to label all glassware and vials.
- 9.8. Always check the sample ID when transferring or vialing samples and extracts, or when changing glassware.
- 9.9. Constant attention must be given to extracts when they are on the water bath or turboVap. Check extracts often when solvent levels are low to ensure extracts do not concentrate to dryness.
- 9.10. Review the Special Analytical Requirements Form (3002F), etc.) prior to extracting samples to determine if special procedures are required.
- 9.11. The LIMS Data entry person will review all Extraction Requirement sheets (Form 3002F) and bench sheets before distribution.
- 9.12. All logbooks are reviewed monthly for completeness and accuracy by the laboratory supervisor





9.13. The QA section will periodically review the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

## 10. Calibration and Standardization

10.1. Not Applicable

## 11. Procedure

11.1. Review the Special Analytical Requirements form prior to extracting samples to determine if special procedures are required.

11.2. Fill out a bench sheet for the job.

11.3. Warm the samples to room temperature.

11.4. Extraction:

11.4.1. Mix the sample to ensure that a homogeneous and representative aliquot is analyzed.

Discard any extraneous material (rocks, sticks, etc.) and note this on the Analyst Notes form.

11.4.2. Total solids and pH are analyzed at the time of extraction. See ARI SOP 359S, Sample Screening, for details.

11.4.3. Label each 400 mL beaker with pink label tape containing the following information: ARI job number, sample ID letter, matrix ID, extraction type (HERB) and, on the method blank (MB) and LCS, use the current date as mmddyy.

11.4.4. Prepare two 400 mL beakers with 15 g of anhydrous sodium sulfate for use as the MB and the LCS. Weigh 15 g of sample into each corresponding 400 mL beaker. In a fume hood acidify the samples to pH <2 with approximately 1 mL (1/4 pipette) of concentrated HCl and stir with a glass rod. Add 15-30 g of anhydrous sodium sulfate to the soil samples and mix well. The samples should have a sandy consistency.

11.4.5. Add surrogates to each sample, MB, LCS and MS as specified on the Extraction Special Requirement Form (3002F). In addition to the surrogates, the LCS and MS receive a full list herbicide spike. To verify that each surrogate and spike addition is accurate, each will be witnessed and documented by another laboratory technician.

11.4.6. After the surrogate and spike solutions have been added, immediately pour approximately 90 mL of 7:3 methylene chloride/acetone into each sample.

11.4.7. Sonicate the samples as follows: place the sonic probe into the sample beaker with the tip below the surface of the solvent but above the soil layer. Set the timer for 3 minutes, the mode to 'pulse', and the duty cycle to 50%.

11.4.8. Once the timer shuts off, remove the beaker, and pour the extraction solvent through the 100 mm glass funnel into a labeled Kurderna-Danish flask.



11.4.9. Repeat steps sonication two more times using approximately 90 mL of 70:30 hexane/methylene chloride each time.

11.5. Hydrolysis:

11.5.1. To 35ml of organic free water add approximately 5 mL of 37% KOH solution. Add this solution and 4-5 boiling chips to each Kuderna-Danish flask. Attach a three ball Snyder column to the flask and place on a water bath set at 100°C.

11.5.2. Periodically, swirl the flask to enhance the solvent evaporation. After the initial concentrating, about 15 minutes, remove the Snyder column and completely evaporate the remaining solvent until no solvent vapors are escaping flask, about 30 – 60 minutes total.

11.5.3. When all the solvent has evaporated, remove the flask from the water bath and allow cooling.

11.6. Clean-up & Back Extraction:

11.6.1. Pour the basic extract (water) from the Kuderna-Danish flask into a 250 mL separatory funnel which has been labeled with the sample ID. Rinse the flask two times with approximately 20ml of deionized water and add the rinsate to the corresponding separatory funnel.

11.6.2. Verify that the pH of the extract is >12. Dip the pointed end of a disposable 5-<sup>3</sup>/<sub>4</sub>" pipette into the extract and verify with broad range pH paper. Add 10 N NaOH solution, drop by drop if necessary, and note this on the Analyst Notes form.

11.6.3. Add 60 mL of methylene chloride to the sample. Seal and shake the separatory funnel vigorously for two minutes, periodically venting. Allow the organic phases to separate. Repeat for each sample. Drain the methylene chloride (bottom layer) into a waste beaker. (If an emulsion is present it must be broken up by whatever mechanical means is necessary.) Repeat if the methylene chloride is pulling out any obvious color.

11.6.4. Prepare a 250 ml Erlenmeyer flask for each sample by adding approximately 10g of acidified sodium sulfate.

11.6.5. Acidify the extract to a pH < 2, with 1:1 sulfuric acid solution.

11.6.6. Add 60 mL of 7:3 methylene chloride/acetone to the separatory funnel and shake for two minutes, venting often. Drain the methylene chloride portion (bottom layer) into the corresponding 250 mL Erlenmeyer flask containing sodium sulfate. Extract the water two more times with 60 mL of 7:3 methylene chloride/hexane. The bottom layer should be drained into the Erlenmeyer flask containing sodium sulfate.

11.6.7. Discard the water and transfer the label to the 250 mL Erlenmeyer flask containing the extract. Additional anhydrous sodium sulfate may be added to the extract in a sufficient



quantity so that some sulfate remains “free flowing”. Allow the extract to stand for 1 hour to remove any water in the extract.

- 11.7. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporation flask. Add 2-3 boiling chips and a blue clip.
- 11.8. Pour the extract into the 500 mL KD evaporator flask being careful not to pour any sodium sulfate into the flask. Rinse the Erlenmeyer flask once with 40ml of hexane and add this to the KD as well. Transfer the label with the sample ID to the K-D. Repeat for each sample.
- 11.9. Attach a three ball Snyder column to the KD, and put the entire apparatus on a water bath set at approximately 100° C. Wet the inside of the Snyder column with 1-2 mL of hexane before boiling starts.
- 11.10. Once the extract has concentrated to 4-8 mL, remove the apparatus from the water bath, and place in a cooling rack for about 10 minutes.
- 11.11. Remove the Snyder column, and remove all water from the joint between the flask and the concentrator tube with a Kimwipe. Disassemble the tube and flask. Transfer the label with the sample ID to the tube.
- 11.12. Using a Pasteur pipet, transfer the extract to a 15 mL disposable centrifuge tube, adjust the volume to 10.0 ml, and cap with a Teflon lined phenolic screw cap. Transfer the sample label with the sample ID and deliver samples to the GC lab for esterification and chromatographic analysis.

## 12. Data Analysis and Calculations

- 12.1. The Organic Extractions’ Supervisor or Lead will review all bench work and bench sheets before distribution.
- 12.2. Review project documentation (special analytical requirements, etc), prior to extracting samples to determine if special procedures are required.
- 12.3. The QA section will periodically review bench sheets to verify that the documentation is clear and accurate.

## 13. Method Performance

- 13.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.
- 13.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

## 14. Pollution Prevention



- 14.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are
- 14.2. Pour any residual wastewater into the container in the Water Extraction Lab fume hood.
- 14.3. Do not discard organic solvent into the sink. Pour all waste solvent into the labeled 55-gallon drum located in the Hazardous Waste Storage Area.
- 14.4. Disposed expired standards into the designated barrel in the hazardous waste room.
- 14.5. Samples that are designated as hazardous waste by the LIMS “Hazardous Report” must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

## **15. Data Assessment and Acceptance Criteria for Quality Control Measures**

- 15.1. Not Applicable

## **16. Corrective Actions for Out of Control Events**

- 16.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.
- 16.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Appendix 20.1

## **17. Contingencies for Handling Out-of-Control or Unacceptable Data**

- 17.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a ‘Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

## **18. Waste Management**

- 18.1. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled “Solvent Contaminated Solids” located in the Hazardous Waste Room when the accumulation pails are full.



- 18.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area
- 18.3. Disposed expired standards into the designated barrel in the hazardous waste room.
- 18.4. Place samples that designate as hazardous using the LIMS "Hazardous Report" in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.
- 18.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.
- 18.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

## 19. Method References

- 19.1. SW-846, "Separatory Funnel Liquid-Liquid Extraction", Method 3510-C, Revision 3, December, 1996. USEPA Test Methods for Evaluating Solid Waste, (SW-846, 3rd Edition). Appendices

## 20. Appendices

- 20.1. Corrective Action Charts



**Analytical Resources, Incorporated**  
Analytical Chemists and Consultants

## **Standard Operating Procedure**

### **Extraction of Water Samples for Organophosphorous Pesticides (EPA Method 8141)**

**SOP 326S  
Revision 008**

**Revision Date: 8/11/06  
Effective Date: 8/11/06**

Prepared By:

Jim Hawk

Approvals:

Sam N. Behe

Laboratory / Section Manager

David R. Mitchell

Quality Assurance





## 1. Scope and Application

- 1.1. This method details the extraction of water samples by separatory funnel in preparation for organophosphorous pesticides analysis. The SOP is written to meet the requirements of SW-Method 3510C.

## 2. Definitions

- 2.1. ECD (Electron Capture Detector) - Sensitive to organic molecules with highly electronegative functional groups. Especially suitable for detecting PCB's (polychlorinated biphenyls) and organochlorine pesticides.
- 2.2. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.
- 2.3. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example< to determine the effect of the sample matrix on a method's recovery efficiency.
- 2.4. Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 2.5. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 2.6. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

## 3. Interferences

- 3.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.
- 3.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.
- 3.3. Some lots of methylene chloride may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have is verified free of contaminates





- 3.4. Laboratory supplies and equipment are potential sources of interfering contamination
  - 3.4.1. Make sure items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.
  - 3.4.2. Use glassware that is thoroughly cleaned, kiln fired (SOP # 301S) and solvent rinsed.
- 3.5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.
- 3.6. Standard solutions or solvents must not contain extraneous compounds or other chemical interferences. All standards are verified by GCMS/GC/ECD prior to use. All solvent lots and support materials are checked for purity prior to use.
- 3.7. Naturally occurring non-petroleum organic material may contribute to biogenic interferences. Acid/silica gel clean-up can be used to assist in the reduction or elimination of these interferences.

#### **4. Safety**

- 4.1. The toxicity or carcinogenicity of each reagent used in this SOP is not been precisely defined. Treat each chemical compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.
- 4.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection
- 4.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT).
- 4.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted as the need may arise. The MSDS file is located in the central project management area. MSDS are also available online, at <http://hazard.com/MSDS/>.
- 4.5. Environmental Samples may contain hazardous waste; treat them as potential health hazards.
- 4.6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be stored for repair.
- 4.7. Use nitrile (blue) gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride.
- 4.8. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware. Do not glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be set aside for repair.



## 5. Equipment and Supplies

### 5.1. Equipment

- 5.1.1. Personal protective equipment (PPE)
  - 5.1.1.1. Gloves
  - 5.1.1.2. Laboratory coat
  - 5.1.1.3. Goggles
- 5.1.2. Nitrogen evaporator, N-Evap or equivalent
- 5.1.3. PH meter, Checker by HANNA or equivalent
- 5.1.4. Glass etcher / engraver
- 5.1.5. Water Bath, Precision Scientific51220042 or equivalent
- 5.1.6. Zymark TurboVap LV (Set at 40°C)
- 5.1.7. Centrifuge Beckmann GP or equivalent
- 5.1.8. Hamilton gastight syringes: 50µL, 250µL, 1000µL

### 5.2. Glassware

- 5.2.1. Graduated cylinder, glass, pre-cleaned graduated 'to deliver'. One 100, 1000 or 2000 mL cylinder per sample depending on volume of ample to be extracted
- 5.2.2. Separatory funnel (SF), glass with PTFE stopper and stopcock plug (one for each sample)
  - 5.2.2.1. 2000 mL SF for 1 Liter samples
  - 5.2.2.2. 1000 mL SF for 500 mL samples
  - 5.2.2.3. 250mL SF for 100 mL (TCLP) samples
- 5.2.3. Erlenmeyer flask, Glass, 250 mL (one for each sample)
- 5.2.4. Centrifuge bottles, glass, 200 mL
- 5.2.5. Powder Funnel, glass, 70mm
- 5.2.6. Kuderna-Danish concentrating apparatus, glass, single walled with 10mL concentrator tube, 500mL concentrator flask and three-ball Snyder Column
- 5.2.7. Turbo Tube, glass, 60 mL with 0.5 mL reservoir
- 5.2.8. 'Tilt-a-pet' dispenser, glass, 30 or 60 mL used to add a measured volume of extracting solvent to the sample in a separatory funnel
- 5.2.9. Drying column, glass, 25 x 340 mm
- 5.2.10. Vial auto sampler clear glass, 2.5mL with PTFE lined crimp cap.
- 5.2.11. Pasteur Pipette, borosilicate glass, 5 ¼ inch, disposable
- 5.2.12. Centrifuge tube, borosilicate glass, 15mL.



### 5.3. Supplies

- 5.3.1. Boiling Chips – PTFE (Chemware)
- 5.3.2. Glass wool, neutral, prepared by heating at 400 °C for 4 hours
- 5.3.3. GPC Medium, Biobeads S-X3 (200-400 mesh) or equivalent 75 g for each GPC column
- 5.3.4. Wide Sharpie Permanent Pen
- 5.3.5. Brown label tape
- 5.3.6. Kim wipe

## 6. Reagents and Standards

- 6.1. Hexane, high purity
- 6.2. Methylene Chloride, high purity
- 6.3. Concentrated sulfuric acid
- 6.4. 1:1 sulfuric acid solution is prepared by adding 500mL of sulfuric acid to 500mL of organic free water
- 6.5. 10 N Sodium Hydroxide solution
- 6.6. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours
- 6.7. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.
- 6.8. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (form 10076F)

## 7. Sample Collection, Preservation, Shipment and Storage

- 7.1. Store all water samples at 2°C to 6°C. Warm the samples to ambient temperature prior to extraction.
- 7.2. Required holding time for all water samples is 7 days from sample collection. Extract aqueous samples within seven days of collection. When holding times are or may be compromised inform your supervisor immediately
- 7.3. TCLP and SPLP extracts are considered water samples with a 7 holding time.
- 7.4. Some samples are shared with the metals and or conventionals laboratories. These samples are placed in a share bin in Refrigerator 5. SOP 1019S includes procedures for handling shared samples.

## 8. Quality Control

- 8.1. One method blank will be extracted for each batch of 20 or fewer samples.



- 8.2. One LCS (spike blank) will be extracted for each batch of 20 or fewer samples.
- 8.3. For TCLP, one matrix spike will be extracted for each batch of 20 or fewer samples.
- 8.4. Upon client's request one MS/MSD will be extracted for each batch of 20 or fewer samples, providing sufficient sample is available for the analysis. If insufficient volume is received for MS/MSD for each batch of 20 samples, a LCS Duplicate will be extracted.
- 8.5. To verify that surrogate and matrix spiking is accurate, spiking will be witnessed and documented by a second laboratory technician.
- 8.6. For the procedure, take things slowly and carefully and write down any noted problems or deviations on the Analyst Notes Form (Form 3056F). Even if you believe the deviation will not affect the final result, you may not be aware of how dramatically some of these changes may affect surrogate and matrix spike recoveries. If you run into technical problems that cannot be answered by your supervisor, notify the GC supervisor or the senior chemist immediately for instructions.
- 8.7. Use a permanent marker to label all glassware and vials.
- 8.8. Always check the sample ID when transferring or vialing samples and extracts, or when changing glassware.
- 8.9. Constant attention must be given to extracts when they are on the water bath or turboVap. Check extracts often when solvent levels are low to ensure extracts do not concentrate to dryness.
- 8.10. Review project documentation (special analytical requirements, etc.) prior to extracting samples to determine if special procedures are required.
- 8.11. The LIMS Data entry person will review all Extraction Requirement sheets (Form 3002F) and bench sheets before distribution.
- 8.12. All logbooks are reviewed monthly for completeness and accuracy by the laboratory supervisor
- 8.13. The QA section will periodically review the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

## 9. Calibration and Standardization

- 9.1. Verify the accuracy of the pH meter using standard buffers at pH 4, 7, and 10.

## 10. Procedure

- 10.1. Review the Special Analytical Requirements (Form 3002F) form prior to extracting samples to determine specific extraction requirements.
- 10.2. Fill out bench sheet for job. (See attached example.)



- 10.3. Allow the samples to equilibrate to ambient temperature prior to extraction..
- 10.4. Label each separatory funnel with a piece of brown label tape with the following information: job number, sample ID letter, matrix ID, type of extraction (OP-Pest), and, for the method blank and the LCS, the date.
- 10.5. Use a graduated cylinder to measure the volume of sample required on Form 3220F (500 or 1000mL), and transfer it to a separatory funnel. Add 30 mL (60 mL for a 1000 mL sample) methylene chloride to each empty sample bottle and swirl. Add this rinse solvent to the graduated cylinder and rinse it. Add the rinsate to a separate 250mL Ehrlenmeyer flask labeled for each sample. Repeat for each sample. Use a clean cylinder for each sample. Notify the Extractions Supervisor immediately when there is insufficient sample to measure the volume required on Form 3002F.
- 10.6. Prepare two separatory funnels with a volume of deionized water equal to the sample volumes. These are used for the method blank LCS analyses
- 10.7. Measure the pH of the samples with a pH meter. It should be between pH 6-8. If necessary, adjust the pH with either 1:1 sulfuric acid solution or 10 N sodium hydroxide solution. Note action on Analyst Notes form. Record the pH on the bench sheet.
- 10.8. Add the volume of surrogate solution required on Form 3002F to each sample, the method blank and the LCS. To verify that the surrogate spiking is accurate, surrogate addition will be witnessed and documented by a second laboratory technician.
- 10.9. Add the volume of matrix spike solution required on Form 3002F to each sample and the LCS. To verify that the matrix spike addition is accurate, spike addition will be witnessed and documented by a second laboratory technician.
- 10.10. Add the methylene chloride rinse from the 250mL Ehrlenmeyer flask to the corresponding sample in it's separatory funnel.
- 10.11. Seal and shake each separatory funnel vigorously for 2 minutes, periodically venting to release pressure in the separatory funnel.
- 10.12. Allow organic and aqueous layers to separate. Drain entire organic layer (including any emulsion) into the corresponding Erlenmeyer flask.
- 10.13. Add 30 mL (60 mL for a 1000 mL sample) of methylene chloride to each separatory funnel, and then repeat Sections 10.11 and 10.12 two more times. When an emulsion is present, it must be broken up by whatever mechanical means necessary until all the non-organic material (water, silt, etc.) is removed from the extract. Drain the organic layer into the corresponding 250mL Erlenmeyer flask. After the final organic layer has been drained, dry the extract with



- enough anhydrous sodium sulfate so that the anhydrous sodium sulfate is “free flowing”. Transfer the label tape to the corresponding 250mL Erlenmeyer flask.
- 10.14. Assemble a Kuderna-Danish (K-D) concentrator by attaching a methylene chloride rinsed 10mL concentrator tube to a methylene chloride rinsed 500mL evaporation flask. Add 2-3 clean boiling chips. Prepare a drying column by placing a glass wool plug inside at the narrow end and filling the column with anhydrous sodium sulfate to a bed height of approximately 5cm. Rinse the prepared column with 30mL methylene chloride.
  - 10.15. Verify that the anhydrous sodium sulfate is “free flowing”. If it not, add more anhydrous sodium sulfate and wait 5-10 minutes before proceeding. Dry the extract by passing it through the drying column and collecting it in the K-D concentrator. Rinse the Erlenmeyer flask three times with methylene chloride and add this to the column as well. When the entire extract has eluted rinse the drying column with 15mL methylene chloride. Transfer the label to the K-D.
  - 10.16. Remove drying column and discard the anhydrous sodium sulfate to the waste buckets marked for halogenated waste. Attach a three ball Snyder column, and put entire apparatus on water bath. Wet the inside of the Snyder column with 1-2mL of methylene chloride before boiling starts.
  - 10.17. Remove the Snyder column, remove all water from the joint between the flask and the concentrator tube with a Kimwipe, and disassemble the tube and flask. Transfer the labels with sample ID to a methylene chloride rinsed pre-labeled turbo tube with a 0.5mL reservoir. Pour the extracts from the K-D apparatus into the corresponding turbo tube. Rinse the K-D apparatus with 5-10 mL methylene chloride and add the rinsate to the turbo tube.
  - 10.18. Remove the labels from the turbo tube. Verify the turbo tube was previously labeled at the top with a wide permanent pen. Mount the turbo tubes onto the Turbo Vap tray and place inside the Turbo Vap set at 40 °C. The water inside the turbovap must be as high as the level of the extract. Select the row of Nitrogen nozzles to be used by pressing the row panel on the Turbo Vap. Place an empty 60mL VOA vial under the nozzles not being used in the selected row to prevent splashing. Set the timer on the turbovap for 5 minutes initially. (Analyst discretion is advised). Close the lid of the Turbo Vap and slowly bring the pressure of the nitrogen up to maximum psi by turning the pressure dial clockwise. The extract must not be splashing. Concentrate the extracts to 0.5 mL, it may take additional 1-5 minute intervals after the initial 5 minute setting to complete the concentration. When the concentration is complete, remove the extracts from the TurboVap immediately.
  - 10.19. Concentrate the extract to 1.0 mL (or the final volume specified on Form 3002F)) and filter through a 0.45  $\mu$ m Puradisc filter to remove gross particulate matter. Transfer the extract to a



2.5 mL clear auto sampler vial with PTFE lined crimp cap (labeled with the sample ID) for GC-MS-SIM analysis.

## **11. Data Analysis and Calculations**

11.1. Not Applicable

## **12. Method Performance**

12.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.

12.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

## **13. Pollution Prevention**

13.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are

13.2. Pour any residual wastewater into the container in the Water Extraction Lab fume hood.

13.3. Do not discard organic solvent into the sink. Pour all waste solvent into the labeled 55-gallon drum located in the Hazardous Waste Storage Area.

13.4. Dispose expired standards into the designated barrel in the hazardous waste room.

13.5. Samples that are designated as hazardous waste by the LIMS "Hazardous Report" must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

## **14. Data Assessment and Acceptance Criteria for Quality Control Measures**

### **15. Corrective Actions for Out of Control Events**

15.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.

15.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Section 22.2.

### **16. Contingencies for Handling Out-of-Control or Unacceptable Data**



16.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a 'Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

## 17. Waste Management

17.1. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled "Solvent Contaminated Solids" located in the Hazardous Waste Room when the accumulation pails are full.

17.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area

17.3. Disposed expired standards into the designated barrel in the hazardous waste room.

17.4. Place samples that designate as hazardous using the LIMS "Hazardous Report" in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.

17.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.

17.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

## 18. Method References

SW-846, "Separatory Funnel Liquid-Liquid Extraction," Method 3510C, Revision 3, December, 1996.





**Analytical Resources, Incorporated**  
Analytical Chemists and Consultants

## **Standard Operating Procedure**

### **Extraction of Soil Samples for Organophosphorous Pesticides (EPA Method 8141)**

**SOP 327S  
Revision 007**

**Revision Date: 7/21/06  
Effective Date: 7/21/06**

Prepared By:

Jim Hawk

Approvals:

Jim N. DeLuca

Laboratory / Section Manager

David R. Mitchell

Quality Assurance





## 1. Scope and Application

- 1.1. This document details the extraction procedure for soils in preparation for organophosphorous pesticide analysis. The SOP is written to meet the requirements of SW-Method 3550B.

## 2. Definitions

- 2.1. ECD (Electron Capture Detector) - Sensitive to organic molecules with highly electronegative functional groups. Especially suitable for detecting PCB's (polychlorinated biphenyls) and organochlorine pesticides.
- 2.2. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.
- 2.3. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the sample matrix on a method's recovery efficiency.
- 2.4. Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 2.5. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 2.6. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

## 3. Interferences

- 3.1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use.
- 3.2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.
- 3.3. Some lots of methylene chloride may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have is verified free of contaminants
- 3.4. Laboratory supplies and equipment are potential sources of interfering contamination



- 3.4.1. Make sure items such as gloves, bench paper, rubber stoppers should not come into contact with samples or extracts.
- 3.4.2. Use glassware that is thoroughly cleaned, kiln fired (SOP # 301S) and solvent rinsed.
- 3.5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.
- 3.6. Standard solutions or solvents must not contain extraneous compounds or other chemical interferences. All standards are verified by GCMS/GC/ECD prior to use. All solvent lots and support materials are checked for purity prior to use.
- 3.7. Naturally occurring non-petroleum organic material may contribute to biogenic interferences. Acid/silica gel clean-up can be used to assist in the reduction or elimination of these interferences.

#### **4. Safety**

- 4.1. The toxicity or carcinogenicity of each reagent used in this SOP is not been precisely defined. Treat each chemical compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.
- 4.2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection
- 4.3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT).
- 4.4. Material Safety Data Sheets (MSDS) that outline hazards, exposure treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted as the need may arise. The MSDS file is located in the central project management area. MSDS are also available online, at <http://hazard.com/MSDS/>.
- 4.5. Environmental Samples may contain hazardous waste; treat them as potential health hazards.
- 4.6. Use nitrile (blue) gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride.
- 4.7. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware. Do not glassware that is chipped, cracked, etched, or scratched. Glassware with minor damage should be set aside for repair.

#### **5. Equipment and Supplies**

##### 5.1. Equipment

##### 5.1.1. Personal protective equipment (PPE)



- 5.1.1.1. Gloves
- 5.1.1.2. Laboratory coat
- 5.1.1.3. Goggles
- 5.1.2. Nitrogen evaporator, N-Evap or equivalent
- 5.1.3. Water Bath, Precision Scientific 51220042 or equivalent
- 5.1.4. Zymark TurboVap LV (Set at 40°C)
- 5.1.5. UltraSonic Mixer – Branson 450 with ¾” titanium horn
- 5.1.6. Drying oven (VWR BIOE) set at 105 C (±5)
- 5.1.7. Analytical Balance- Top loading, readable to 0.02g
- 5.1.8. Centrifuge Beckmann GP or equivalent
- 5.1.9. Hamilton gastight syringes: 50µL, 250µL, 500µL, 1000µL
- 5.2. Glassware
  - 5.2.1. Erlenmeyer flask, Glass, 5000 mL (one for each sample)
  - 5.2.2. Centrifuge bottles, glass, 200 mL
  - 5.2.3. Beaker, glass, 400mL (one for each sample)
  - 5.2.4. Beaker, glass, 100mL (one for each sample)
  - 5.2.5. Powder Funnel, glass, 100 mm
  - 5.2.6. Kuderna-Danish concentrating apparatus, glass, single walled with 10mL concentrator tube, 500mL concentrator flask and three-ball Snyder Column
  - 5.2.7. Turbo Tube, glass, 60 mL with 0.5 mL reservoir
  - 5.2.8. ‘Tilt-a-pet’ dispenser, glass, 30 or 60 mL used to add a measured volume of extracting solvent to the sample in a separatory funnel
  - 5.2.9. Drying column, glass, 25 x 340 mm
  - 5.2.10. Vial auto sampler clear glass, 2.5mL with PTFE lined crimp cap.
  - 5.2.11. Pasteur Pipette, borosilicate glass, 5 ¾ inch, disposable
  - 5.2.12. Glass stir rod
- 5.3. Supplies
  - 5.3.1. Spatula, stainless steel
  - 5.3.2. Weighing dish, Aluminum
  - 5.3.3. Filter Medium, 0.45µm, Whatman Puridisc ® (13mm PTFE) or equivalent
  - 5.3.4. Boiling Chips – PTFE (Chemware)
  - 5.3.5. Glass wool, neutral, prepared by heating at 400 °C for 4 hours
  - 5.3.6. Wide Sharpie Permanent Pen
  - 5.3.7. Brown label tape



#### 5.3.8. Kim wipe

### 6. Reagents and Standards

- 6.1. Acetone, high purity
- 6.2. Hexane, high purity
- 6.3. Methylene Chloride, high purity
- 6.4. 95% Methylene chloride / 5% Hexane solution
- 6.5. 50% Methylene chloride / 50% Acetone solution
- 6.6. Anhydrous sodium sulfate, prepared by baking in kiln at 400 for 4 hours
- 6.7. Organic Free Water (OFW) – ASTM Type 1 Water (18.6 megaohm resistivity) produced using ARI's central water purification system.
- 6.8. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in the Reagent Preparation Logbook (form 10076F)

### 7. Sample Collection, Preservation, Shipment and Storage

- 7.1. Store all water samples at 2°C to 6°C. Warm the samples to ambient temperature prior to extraction.
- 7.2. Required holding time for all water samples is 7 days from sample collection. Extract aqueous samples within seven days of collection. When holding times are or may be compromised inform your supervisor immediately
- 7.3. TCLP and SPLP extracts are considered water samples with a 7 holding time.
- 7.4. Some samples are shared with the metals and or conventionals laboratories. These samples are placed in a share bin in Refrigerator 5. SOP 1019S includes procedures for handling shared samples.

### 8. Quality Control

- 8.1. One method blank will be extracted for each batch of 20 or fewer samples.
- 8.2. One LCS (spike blank) will be extracted for each batch of 20 or fewer samples.
- 8.3. For TCLP, one matrix spike will be extracted for each batch of 20 or fewer samples.
- 8.4. Upon client's request one MS/MSD will be extracted for each batch of 20 or fewer samples, providing sufficient sample is available for the analysis. If insufficient volume is received for MS/MSD for each batch of 20 samples, a LCS Duplicate will be extracted.
- 8.5. To verify that surrogate and matrix spiking is accurate, spiking will be witnessed and documented by a second laboratory technician.



- 8.6. For the procedure, take things slowly and carefully and write down any noted problems or deviations on the Analyst Notes Form (Form 3056F). Even if you believe the deviation will not affect the final result, you may not be aware of how dramatically some of these changes may affect surrogate and matrix spike recoveries. If you run into technical problems that cannot be answered by your supervisor, notify the GC supervisor or the senior chemist immediately for instructions.
- 8.7. Use a permanent marker to label all glassware and vials.
- 8.8. Always check the sample ID when transferring or vialing samples and extracts, or when changing glassware.
- 8.9. Constant attention must be given to extracts when they are on the water bath or turboVap. Check extracts often when solvent levels are low to ensure extracts do not concentrate to dryness.
- 8.10. Review project documentation (special analytical requirements, etc.) prior to extracting samples to determine if special procedures are required.
- 8.11. The LIMS Data entry person will review all Extraction Requirement sheets (Form 3002F) and bench sheets before distribution.
- 8.12. All logbooks are reviewed monthly for completeness and accuracy by the laboratory supervisor
- 8.13. The QA section will periodically review the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

## **9. Calibration and Standardization**

- 9.1. Verify the accuracy of the pH meter using standard buffers at pH 4, 7, and 10.

## **10. Procedure**

- 10.1. Review the Special Analytical Requirements Form 3002F (pink sheet) prior to extracting samples to determine if special procedures are required.
- 10.2. Percent total solids and pH are analyzed at time of OP-Pest analysis using the procedure in Sample Screening SOP 359S.
- 10.3. Prepare a bench sheet for job (see attached example).
- 10.4. Allow the samples to equilibrate at ambient temperature. Mix samples to ensure a homogenous and representative aliquot is used. Discard any extraneous materials (rocks, sticks, etc.) and note action on analyst notes.



- 10.5. Label a 100 mL beaker for each sample with brown label tape containing the following information: job number, sample ID letter, matrix ID, extraction type (OP-Pest), and for the method blank and the LCS, the date.
- 10.6. Using a top loading balance, prepare two 100 mL beakers with 10g of anhydrous sodium sulfate for use as a method blank and an LCS. Weigh 10g of sample into each corresponding 400 mL beaker. Add 5 to 20 g of anhydrous sodium sulfate to the soil samples and mix well. The samples should have a sandy consistency.
- 10.7. Consult Special Requirements Form 3002F and add the required volume of surrogate solution to each sample, the method blank and the LCS. To verify that the surrogate spiking is accurate, surrogate addition will be witnessed and documented by another laboratory technician. If the sample is an LCS or matrix spike, proceed to Section 10.8. After the surrogate, spike, and sodium sulfate have been added, immediately pour approximately 30 mL of 1:1 methylene chloride/acetone into each sample.
- 10.8. Following Special Requirements form 3002F add the required volume of full list spiking solution to each LCS.
- 10.9. Following Special Requirements form add the required volume of matrix spike solution all matrix spike samples. To verify that matrix spiking is accurate, spike addition will be witnessed and documented by another laboratory technician.
- 10.10. Sonicate the sample as follows: Place the sonic probe into the sample beaker with the tip below the surface of the solvent but above the sediment layer. Set the timer for 3 minutes, the power output to 10, the mode to 'pulse' and the duty cycle to 50%.
- 10.11. Once the timer shuts off, remove the beaker and pour the extraction solvent through a 100 mm powder funnel, with a glass wool plug holding approximately 5 g  $\text{Na}_2\text{SO}_4$ , into a 500 mL Erlenmeyer flask which has been labeled with the sample ID.
- 10.12. Repeat Sections 6.10 and 6.11 two more times. Use 100% Methylene chloride for the third extraction
- 10.13. Following sonication, the entire extract is poured through the funnel, the beaker is rinsed, the rinsate poured through the funnel. Rinse the funnel and the extracted sample with Methylene chloride. All rinses are collected in the flask. Transfer the label with sample ID to the flask.
- 10.14. Assemble a Kuderna-Danish (K-D) concentrator by attaching a methylene chloride rinsed 10 mL concentrator tube to a rinsed 500 mL evaporation flask. Prepare a drying column, one for each sample, by putting a glass wool plug inside at the narrow end and filling the column with anhydrous sodium sulfate to a bed height of approximately 10 cm. Rinse the prepared column once with 30 mL methylene chloride.





- 10.15. Dry the extract by passing it through the drying column and collecting it in the K-D concentrator. Rinse the Erlenmeyer flask three times with methylene chloride and add this to the column as well. Once the entire extract has passed through the drying column rinse the column with methylene chloride. Transfer the label with the sample ID to the K-D. Check the sample IDs.
- 10.16. Remove drying column, add 2 - 3 clean boiling chips to the concentrator flask, attach a methylene chloride rinsed three ball Snyder column, and put entire apparatus on a water bath. Wet the inside of the Snyder column with 1 - 2 mL of methylene chloride before boiling starts.
- 10.17. Once the extract reaches 4 - 6 mL, the solvent is exchanged to hexane by flushing the Snyder column with approximately 15 mL of 95% Methylene / 5% Hexane. Mark an X on the label to indicate the exchange process has taken place
- 10.18. Once the extract has concentrated to about 5-10 mL, remove the apparatus the water bath. Place the apparatus in a cooling rack and allow it to stand for about 10 minutes.
- 10.19. Remove the Snyder column, from the K-D apparatus. Transfer the labels with sample ID to a methylene chloride rinsed pre-labeled turbo tube with a 0.5mL reservoir. Pour the extracts from the K-D apparatus into the corresponding turbo tube. Rinse the K-D apparatus with 5-10 mL methylene chloride and add the rinsate to the turbo tube.
- 10.20. Remove the labels from the turbo tube. Verify the turbo tube was previously labeled at the top with a wide permanent pen. Mount the turbo tubes onto the Turbo Vap tray and place inside the Turbo Vap set at 40 °C. The water inside the turbovap must be as high as the level of the extract. Select the row of Nitrogen nozzles to be used by pressing the row panel on the Turbo Vap. Place an empty 60mL VOA vial under the nozzles not being used in the selected row to prevent splashing. Set the timer on the turbovap for 5 minutes initially. (Analyst discretion is advised). Close the lid of the Turbo Vap and slowly bring the pressure of the nitrogen up to maximum psi by turning the pressure dial clockwise. The extract should not be splashing. Concentrate the extracts to 0.5 mL, it may take 1-5 minute intervals after the initial 5 minute setting to complete the concentration. When the concentration is complete, remove the extracts from the TurboVap immediately.
- 10.21. Concentrate the extract to 1.0 mL (or another specified final volume) and filter through a 0.45 µm Puradisc filter to remove gross particulate matter. Transfer the extract to a 2.5 mL clear auto sampler vial with PTFE lined crimp cap (labeled with the sample ID) for GC-MS-SIM analysis.

## 11. Data Analysis and Calculations

- 11.1. Not Applicable



## 12. Method Performance

- 12.1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.
- 12.2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

## 13. Pollution Prevention

- 13.1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are
- 13.2. Pour any residual wastewater into the container in the Water Extraction Lab fume hood.
- 13.3. Do not discard organic solvent into the sink. Pour all waste solvent into the labeled 55-gallon drum located in the Hazardous Waste Storage Area.
- 13.4. Disposed expired standards into the designated barrel in the hazardous waste room.
- 13.5. Samples that are designated as hazardous waste by the LIMS “Hazardous Report” must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

## 14. Data Assessment and Acceptance Criteria for Quality Control Measures

### 15. Corrective Actions for Out of Control Events

- 15.1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.
- 15.2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Section 22.2.

### 16. Contingencies for Handling Out-of-Control or Unacceptable Data

- 16.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a ‘Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

## 17. Waste Management



- 17.1. Place the solids in the designated 5-gallon "satellite accumulation stations" located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled "Solvent Contaminated Solids" located in the Hazardous Waste Room when the accumulation pails are full.
- 17.2. Discard all waste solvent (Hexane and Methylene Chloride) into the 55 gallon drum labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area
- 17.3. Disposed expired standards into the designated barrel in the hazardous waste room.
- 17.4. Place samples that designate as hazardous using the LIMS "Hazardous Report" in the designated drum in the Hazardous Waste Storage Area when they are disposed. SOP 1003S describes the process for disposal of samples. Excess extracts and expired spiking solutions must be disposed of in the container labeled "Chlorinated Solvents" located in the Hazardous Waste Storage Area.
- 17.5. ARI's Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.
- 17.6. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

## 18. Method References

- 18.1. U.S. EPA, "Sonication Extraction", (SW-846), Method 3550B, Revision 3, December, 1996.



**Analytical Resources, Incorporated**  
Analytical Chemists and Consultants

## Standard Operating Procedure

### Chlorinated Herbicide Analysis EPA Method 8151A

**SOP 405S**  
**Revision 008**

**Revision Date: 7/18/06**  
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Quality Assurance Standard Operating Procedure





## Chlorinated Herbicide Analysis – EPA Method 8151A

### 1. Scope and Application

- 1.1. This document summarizes ARI's procedures to detect and quantify selected chlorinated herbicides in organic solvent extracts of environmental aqueous and soil samples.
- 1.2. These procedures are restricted to use by, or under close supervision of an analyst experienced in the use of gas chromatography and the interpretation of gas chromatographs.
- 1.3. Routine analytes and reporting limits are:

		Reporting Limits			
		Aqueous Samples		Solid Samples	
Sample Weight / Volume:		500 mL	500 mL	15 g	15 g
Extract Final Volume:		10 mL	50 mL	10 mL	50 mL
Compound Name	CAS No.	µg/L	µg/L	µg/Kg	µg/Kg
2,4-D	94-75-7	0.20	1.00	6.67	33.3
2,4-DB	94-82-6	0.20	1.00	6.67	33.3
2,4,5-TP (Silvex)	93-72-1	0.05	0.25	1.67	8.33
2,4,5-T	93-76-5	0.05	0.25	1.67	8.33
Dalapon	75-99-0	0.20	1.00	6.67	33.3
Dicamba	1918-00-9	0.10	0.50	3.33	16.7
Dichloroprop	120-36-5	0.20	1.00	6.67	33.3
Dinoseb	88-85-7	0.05	0.25	1.67	8.33
MCPA	11100-14-4	50.00	250.00	1670	8330
MCPP		50.00	250.00	1670	8330
PCP		0.05	0.25	1.67	8.33
2,4-DCPA		0.25	1.25	8.33	41.7

### 2. Summary of Procedure

- 2.1. Extracted herbicide acids are methylated using Diazomethane followed by analysis of the esterified acids using (GC-ECD). The concentration of detected analytes is determined by comparison with herbicide in solution at known concentrations. Prior to analysis the GC-ECD instrument is optimized and calibrated. The resulting GC-ECD data is reviewed, compared with



historic data, and verified by the analyst before it is forwarded to ARI's project management team for delivery to a client.

### 3. Definitions

- 3.1. ECD (Electron Capture Detector) - Sensitive to organic molecules with highly electronegative functional groups. An ECD is especially suitable for detecting PCB's (polychlorinated biphenyls), organochlorine pesticides and chlorinated herbicides.
- 3.2. Laboratory Control Sample (LCS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.
- 3.3. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the sample matrix on a method's recovery efficiency.
- 3.4. Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 3.5. Method Blank (MB) – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 3.6. Surrogate – Surrogates are compounds chemically similar to the targeted analytes, but not expected to be found in environmental samples used to evaluate extraction efficiency by measuring recovery. Surrogates are added to every sample analyzed.

### 4. Interferences

- 4.1. High purity reagents must be used to minimize interference problems.
- 4.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination (if needed).
- 4.3. The derivatization process and ether solvent may cause interferences that can mask or distort the Dalapon peak. It is critical that this peak be correctly integrated.



- 4.4. Organic acids, especially chlorinated acids, cause the most direct interference with herbicide concentration determinations. Phenols, including chlorophenols, may also interfere with this procedure.
- 4.5. Alkaline hydrolysis and subsequent extraction of the basic solution should remove many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with GC-ECD analysis.
- 4.6. Herbicides, being strong organic acids, react readily with alkaline substances. Therefore, acid rinsed glassware and glass wool must be used.

## 5. Safety

- 5.1. The toxicity and carcinogenicity of each reagent used in this method is not precisely defined. However, all compounds and solutions should be treated as health hazards, and exposure of these chemicals to skin and clothing should be minimized to the lowest possible level by whatever means available.
- 5.2. Skin contact with all chemicals should be minimized by the used of nitrile gloves, safety glasses, and laboratory coats.
- 5.3. All materials should be handled in the fume hoods to avoid exposure to fumes.
- 5.4. Special care should be taken when derivatizing samples for analysis. The diazomethane produced by the reaction of diazald, sodium hydroxide, and methanol is both toxic and explosive. The methylation MUST take place within a fume hood, and the analyst should work with the sash down if possible. Avoid heating the solution above 90 degrees centigrade, or exposing it to alkali metals, as explosion may result.
- 5.5. ARI maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Consult with MSDS sheets for all chemicals handled.

## 6. Equipment and Supplies

- 6.1. Gas chromatograph
  - 6.1.1. Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories including:
  - 6.1.2. autosampler
  - 6.1.3. analytical columns Fused-silica capillary columns - One of the two following column pairs are used. Columns used may vary depending on currently available technology (other column pairs may be used so long as they generate equivalent data):





- 6.1.4. Column 1: DB-5, 30 m, 0.53 mm ID, 1.5  $\mu\text{m}$  film/Column 2: DB608, 30 m, 0.53 mm ID, 0.5  $\mu\text{m}$  film.
- 6.1.5. Column 1: Rtx-CLPesticides, 30 m, 0.53 mm ID, 0.50  $\mu\text{m}$  film/Column 2: Rtx-CLPesticides II, 30 m 0.53 mm ID, 0.42  $\mu\text{m}$  film.
- 6.1.6. y-connectors
- 6.2. gases
- 6.3. ECD detectors
- 6.4. Data system - A computer system must be interfaced to the GC. The system must allow the recording of instrument response as a function of time
- 6.5. The data must be securely stored for at least two years from date of acquisition.
- 6.6. Syringes - 10  $\mu\text{L}$ , 25  $\mu\text{L}$ , 50  $\mu\text{L}$ , 100  $\mu\text{L}$ , 500  $\mu\text{L}$ , 1000  $\mu\text{L}$
- 6.7. Volumetric flasks, Class A - 10 mL to 1000 mL.
- 6.8. Bottles - glass with Teflon-lined screw caps or crimp tops.
- 6.9. Balance - Analytical, 0.0001 g.
- 6.10. Autosampler vials- amber 2 ml autosampler vials with crimp caps.
- 6.11. Derivatization apparatus and reagents-
- 6.12. A 60 mL VOA vial, with cap and Teflon lined septum.
- 6.13. A Teflon tube, pushed through the septum.

## 7. Reagents and Standards

- 7.1. Diazald
- 7.2. Methanol.
- 7.3. 3M NaOH in methanol Acetone
- 7.4. Stock Standard Preparation
  - 7.4.1. Prepare stock standard solutions by accurately weighing about 0.025 g of pure material. Dissolve the material in reagent grade hexane or other suitable solvent and dilute to volume in a 25 mL volumetric flask. Larger or smaller volumes can be used at the convenience of the analyst. When compound purity is assayed to be 97% or greater, the weight may be used without correction to calculate the concentration of the stock standard.
  - 7.4.2. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.
  - 7.4.3. Transfer the stock standard solutions into amber bottles with Teflon lined screw-caps. Store at 4°C and protect from light.
  - 7.4.4. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.



- 7.4.5. Stock standard solutions must be re-assayed or replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.
- 7.4.6. Surrogate Stock Standard- The surrogate standard employed is 2,4-Dichlorophenylacetic acid. Prepare a stock solution of this analytes with a concentration of 100 ug/mL by dissolving 10 mg 2,4-Dichlorophenylacetic acid in a small amount of hexane. Transfer to a 100 mL volumetric flask and dilute to volume with methanol.
- 7.4.7. Matrix Spike Stock Standards – The matrix spike standards are Dicamba, 2,4-D, and Silvex (2,4,5-TP). The matrix spike is normally purchased as a commercially certified working solution. If prepared from neat compounds, dissolve 0.1g of material in a small amount of hexane. Transfer to a 100 mL volumetric flask and dilute to volume with methanol
- 7.4.8. Laboratory control samples are spiked with all target analytes. The matrix spike is purchased as a commercially certified working solution.
- 7.5. Working solutions- working standards are prepared from stock standards or purchased as commercially certified mixtures.
- 7.5.1. Working standards expire either on the date of expiration of the stock solutions they are made from, or on the manufacturer's certified expiration date. They must be either re-assayed or replaced at this time.
- 7.5.2. Working standards must be stored at  $4 \pm 2^{\circ}\text{C}$  and protected from light.
- 7.5.3. Working standards should be check frequently for signs of concentration or degradation.
- 7.5.4. Surrogate Working Standards
- 7.5.4.1. Surrogate spiking standard- the surrogate spiking standard is prepared by methylating the 2,4-Dichlorophenylacetic acid surrogate stock such and adjusting the concentration to 2  $\mu\text{g}/\text{mL}$ . This solution is prepared in methanol. This solution is used to spike all extracted samples and QC samples with surrogates.
- 7.5.5. Matrix Spike Working Standards- the matrix spike working standards are used to prepare MS/MSD sets as well as LCSs and LCSDs.
- 7.5.5.1. The matrix spiking standard is prepared by diluting the matrix spiking stock solution in methanol to the appropriate concentrations. Commercially certified solutions are frequently used to prepare the matrix spiking working standards.
- 7.5.6. Calibration working standards- the standards used to prepare calibration curves are usually purchased as commercially certified mixtures.
- 7.5.7. Prepare calibration working standards by diluting these commercially certified standards such that the concentration of each Aroclor is 500 ng/ml.

## 8. Sample Collection, Preservation, Shipment and Storage



- 8.1. Extracts are delivered to refrigerator 17 in the instrument laboratory by extractions technicians.
- 8.2. Analysts in the instrument lab assume custody of the sample extracts and then move them into refrigerator 18.
- 8.3. Extracts must be stored at 4 degrees centigrade ( $\pm 2$  degrees), and protected from light.
- 8.4. Extracts must be analyzed within 40 days of extraction.

## 9. Quality Control

- 9.1. Quality control requirements for initial and continuing calibration are detailed in Section 11 of this document.
- 9.2. Surrogate Standards are added to all samples including any MB, LCS, MS or other QA sample during the extraction process. Surrogate percent recovery is calculated following an initial analysis to determine extraction efficiency.
- 9.3. Method blanks are extracted and analyzed with every batch of twenty or less samples. The purpose of a method blank is to determine the levels of contamination associated with the analytical process.
- 9.4. Laboratory Control Sample(s) are included in each preparation batch to evaluate the accuracy of the analytical method independent of sample matrix-related effects.
- 9.5. Matrix Spike Samples (MS/MSD) may be included in each preparation batch to evaluate the effects of the sample matrix on the analytical process.
  - 9.5.1. As part of a client's QA/QC program, water rinsate samples and/or field/trip blanks (field QC) may accompany soil/sediment samples and/or water samples that are delivered to the laboratory for analysis. The laboratory will not perform MS/MSD analysis on any of the field QC samples.
  - 9.5.2. If a client designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample remaining to perform an MS/MSD, then the laboratory shall choose another sample on which to perform an MS/MSD analysis. At the time the selection is made, the laboratory shall notify the client that insufficient sample was received and identify the sample selected for the MS/MSD analysis. The rationale for the choice of another sample other than the one designated by the client shall be documented in the narrative.
  - 9.5.3. If there is insufficient sample remaining in any of the samples to perform an MS/MSD, the laboratory shall immediately contact the client to inform them of the situation. The client will either approve that no MS/MSD is required, require that a reduced sample aliquot be used for the MS/MSD analysis or request that a LCS/LCSD (See Section 10.3.3) analysis be performed. The laboratory shall document the decision in the narrative.



- 9.5.4. Dilution of MS/MSD extracts to get either spiked compounds or native analytes on scale is not necessary.
- 9.6. Statistical Control- Internal quality control limits for analyte spike and surrogate recoveries and relative percent difference for matrix spike and matrix spike duplicates are statistically generated on a periodic basis.
- 9.6.1. These quality control limits are provided to bench chemists, managers, and QA staff as tools for assessing data quality in real-time at the point of data generation.
- 9.6.2. Practical considerations relating to their dynamic nature require their presentation in a document separate from this SOP. Current control limits may be found in the ARI QAP.
- 9.6.3. All analysts using this SOP must use it in conjunction with Control Limit documentation in order to assess data quality and any possible need for corrective actions.
- 9.7. Initial Demonstration of Proficiency- Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination performed, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat these procedures whenever new staff is trained or significant changes in instrumentation are made. The procedure is outlined in ARI's SOP 1017 – "Training and Demonstration of Proficiency"

## 10. Calibration and Standardization

- 10.1. Prior to sample analysis, the ECD on each column must be calibrated.
- 10.1.1. Calibration Standard Preparation
- 10.1.1.1. The high point of the initial calibration is created by taking an undiluted aliquot of the calibration working solution. This creates an initial calibration standard with concentrations of 100,000 ng/mL for MCPA and MCPP; 2000 ng/mL for 2,4-DB; 400 ng/mL for Dicloroprop, Dalapon, and 2,4-D; 200 ng/mL for Dicamba; 100 ng/mL for Silvex, PCP, Dinoseb, and 2,4,5-T; and 500 ng/mL for the surrogate DCPA.
- 10.1.1.2. The next four calibration standards are prepared by serial dilution. The dilutions are prepared in hexane. The first is prepared by diluting the calibration high point by a factor of two (equal volumes of hexane and calibration standard.)The next three follow the same pattern, diluting each standard by a factor of two. The calibration standards will thus have concentrations of 50,000/1000/250/200/100/50 ng/L, 25,000/500/125/100/50/25 ng/mL, 12,500/250/62.5/50/25/12.5 ng/mL, and 6,250/125/31.25/25/12.5/6.25 ng/mL. The low point is prepared by using a serial dilution, but with a dilution factor of 2.5, for final concentrations of 2,500/50/12.5/10/5/2.5 ng/mL.



- 10.2. Introduce each calibration standard to the GC. All calibration standards, calibration verifications, and samples must use the same instrument parameters. Typically 2 ul injections are used, and this injected volume is split between the two columns using a glass y-connector attached to the guard column and each of the two columns.
- 10.3. For each peak, calculate the Calibration Factor (CF) using the height of the peak and the amount of compound injected (See Section 14.1.)
- 10.4. Calculate the Average CF for each peak (see section 14.2.)
- 10.5. Calculate the Relative Standard Deviation (RSD) for each peak (see section 14.3.)
- 10.6. The average RSD for each compound must be less than or equal to 20%.
- 10.7. Target analytes that exceed 15% RSD will utilize an alternative calibration option. If the analyst determines that there is no detector or chromatographic peak saturation and that the chromatographic system is functioning properly, then the analyst will proceed as follows:
  - 10.7.1. A linear fit calibration that does not include the origin and generates a coefficient of determination ( $R^2$ ) that is greater than or equal to 0.99 is acceptable. ARI will attempt to force calibration curve through zero. Forcing the curve through zero is not the same as including the origin as a point in the calibration. When the curve is forced through zero, the intercept is set to 0 before the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom.
  - 10.7.2. Next the analyst may attempt a quadratic non-linear calibration which may be used with target analytes that have six calibration points only. An additional initial calibration point may be considered for target analytes that require non-linear calibration if only five calibration points are available.
  - 10.7.3. Individual targets that are unable to meet the above requirements must be documented in the case narrative. The initial calibration may still be valid if approved on an individual basis by the Laboratory Supervisor and/or the Laboratory Manager.
  - 10.7.4. See EPA Method 8000C Section 11 for reference to linear fit and non-linear (quadratic) calibration.
- 10.8. Retention time windows- Retention times are established with each initial calibration.
  - 10.8.1. ARI uses a default retention time window of  $\pm 0.05$  minutes. The use of Merlin Microseals (part # 310, Merlin Instrument Co. Half Moon Bay, CA) and Electronic Pressure Control (EPC) maintain exceptionally constant retention times. Retention time studies suggested by the method often result in windows of 0.01 minutes or less.
  - 10.8.2. Laboratory experience working with difficult matrices has shown that narrower retention time windows often lead to false negatives in samples with high background contamination



or very oily matrices. For this reason, the analyst should still use a window of  $\pm 0.05$  minutes to evaluate interferences.

10.8.3. Retention time criteria may change during an analytical sequence if the RT shift is explainable (column maintenance) and CCVs confirm that the calibration is valid. All such deviations must be explained on the Analyst Notes form.

10.9. The instrument initial calibration (ICal) must be verified prior to use with an initial calibration verification (ICV). The ICV standard is prepared using standards independent from the ICal standards. The ICV standard is prepared at the same concentration as the mid-point ICal standard. The ICV standard must have a percent difference  $\leq 15\%$  compared to the ICal midpoint calibration standard or be noted in the analyst's notes as exceeding 15%.

10.10. Continuing Calibration Verification (CCV). The performance of the instrument over time is monitored by the used of continuing calibration verifications.

10.10.1. The CCV standards are prepared from the same source as the ICAL standards. CCV standards are analyzed at the concentration of the midpoint ICAL standard (25,000/500/125/100/50/25 ng/ml).

10.10.2. CCVs must be analyzed at a minimum at the beginning and end of an analytical bracket. Preferably CCVs should be run after every ten samples in an analytical bracket.

10.10.3. No more than 12 hours may pass between CCVs.

10.10.4. The retention times of all analytes in all daily CCVs must fall within the retention time windows established during initial calibration.

10.10.5. Calculate the CF for each analyte in the CCV just as was done with the initial calibration.

10.10.6. The %D between the CCV CF and the initial calibration's average CF must be within  $\pm 15\%$ .

## 11. Procedure

### 11.1. Esterification

11.1.1. Herbicides present in the solvent extracts will be in the free acid form. These acid do not chromatograph well and must be derivatized prior to analysis on the GC/ECD system. Esterification is accomplished by treating the extracts with Diazomethane following the procedure below:

11.1.1.1. CAUTION: Diazomethane is a carcinogen and can explode under certain conditions. Only perform this procedure under a fume hood.

11.1.1.2. \*Do not heat above 90° C-EXPLOSION may result

11.1.1.3. \*Avoid grinding surfaces-EXPLOSION may result



11.1.1.4. \*Store away from alkali metals-EXPLOSION may result

11.1.1.5. \*Solutions of diazomethane will decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

11.1.2. Assemble the diazomethane generator:

11.1.3. With a clean metal spatula place 0.5 to 1 inch of Diazald into a screw top 60 mL glass VOA vial.

11.1.4. Add Methanol to the vial until it is about ½ full (~25 mL).

11.1.5. Add approximately 2 mL of 3M NaOH solution with a disposable pipette and use the pipette end to gently mix the NaOH through the diazald/methanol. The diazomethane gas should start bubbling within a few seconds. If not, add another small amount of NaOH.  
**Caution:** adding too much NaOH will cause the solution to overflow.

11.1.6. Place a Teflon tube through a hole in a Teflon-coated septum top and screw it onto the VOA vial. The Teflon tube is used to purge diazomethane gas directly into each individual sample extract vial received from Organics Extraction.

11.1.7. Allow the diazomethane to bubble into the sample extract until the solvent has a persistent yellow color.

11.1.8. Caution should be taken to not cross contaminate from sample to sample.

11.1.9. Log the reagent number into the Reagent Preparation logbook. .

11.1.10. After derivatization allow the extracts to sit uncapped for five minutes to outgas any unreacted diazomethane.

11.1.11. Store the esterified extracts at  $4 \pm 2^\circ \text{C}$  away from light. Preservation studies indicate that the methylated extracts should be analyzed immediately to minimize transesterification or other chemical reactions.

## 11.2. GC Analysis

11.2.1. The recommended GC operating conditions are as follows (samples must be run using the same instrument conditions as the initial calibration

Initial temperature:	60°C, hold for 1 minute
Temperature program:	60-270°C at 9 C°/min
Final temperature:	270°C, hold for 3 minutes
Injector temperature:	220°C
Detector temperatures:	310°C
Detector make-up gas flows	40 mL/min (front) and 35 mL/min (back)
Column initial flow	3.0 mL/min (hold for 12 minutes)
Column flow ramp	10 mL/min at 1 mL/min



Column flow final hold	5 minutes at 5 mL/min
Injector:	Grob-type, splitless/split
Injector Pulse	20 psi at 0.45 minutes
Split flow	20 mL/min at 0.5 minutes
Sample volume:	1-2 $\mu$ L
Carrier gas:	Helium at 30 cm/sec.

11.3. After the instrument and all extracts have been prepared, the analytical sequence may begin.

11.3.1. Prior to sample analysis the GC system must have an acceptable initial calibration curve (the requirements for the calibration curve are found in Sections 11.2 through 11.9.)

11.3.2. Prior to sample analysis a continuing calibration verification must be analyzed, and this CCV must meet the criteria found in Section 11.11. If time remains in the 12 hour QC period begun with the initial calibration, the midpoint calibration standard from the initial calibration may be used as the CCV provided it meets the requirements found in Section 11.11.

11.4. GC analysis

11.4.1. It is highly recommended that the extract be screened on a GC/ECD using the same type of capillary column. This will minimize contamination of the GC system from unexpectedly high concentrations of organic compounds and may show high background samples that should be analyzed using a medium/high level extraction.

11.4.2. Prepare an aliquot of the derivatized extract.

11.4.3. Analyze the aliquot by GC. The injection volume must be the same volume used for the calibration standards. The recommended GC operating conditions to be used are specified in Section 11.2.

11.4.4. If the response for any target analyte exceeds the response of the analyte in the high concentration initial calibration curve extract dilution must take place. See Section 18 for guidance on dilutions.

11.4.5. Perform all qualitative and quantitative measurements as described in Section 14. Store the extracts at 4°C, protected from light in screw-cap vials equipped with unpierced Teflon lined septa.

11.4.6. After instrumental acquisition is complete, the data must be exported from the GC to the Target data system for data reduction.

11.4.7. After the data has been moved to Target, it must be processed with the proper method. Once the data file is processed, the analyst must review the file in Target. It is essential in order to produce quality data that the analyst ensure that the computer has correctly integrated all peaks and baselines and correctly identified all surrogates and Aroclor peaks.





If corrections are required, the analyst should re-integrate incorrect aspects of the data file. If necessary, the analyst should include magnified chromatogram signals to show manual integrations (raw data printouts will include the chromatogram and will show baseline corrections.)

11.4.8. After a data file has been inspected by the analyst, it must be printed to a paper hardcopy. The analyst must initial and date the target report to sign off on the finished data product, including manual corrections.

11.4.9. The analyst then imports the quantitation numbers into the laboratory information management system (LIMS) so that final reports may be generated. The analyst must turn in for review the ARI job file, which should include copies of all bracketing CVSs, raw data for all the samples and associated QC, the LIMS reports, and an analyst's note form which should include all deviations from procedure or otherwise noteworthy events concerning sample analysis.

## 12. Data Analysis and Calculations

12.1. Tentative identification of any single peak analyte occurs when a peak falls within the retention time window on one of the columns. The analyst should then see if the compound is present on the second column.

12.2. If overlapping or co-eluting peaks preclude identification of an analyte on one or both columns, report the higher of the two compound values and qualify the value with a "Y" flag indicating a raised reporting limit. This flag indicates that the analyte may be present at or below the listed concentration, but second column confirmation was inadequate in the opinion of the analyst. See Appendix 12.2 for data qualifiers.

12.3. If the analyte is identified by the data system in the sample on either column, but the retention time is outside the retention time windows, the analyst should report the higher of the two column values qualified with the "Y" flag. Note: The retention time windows should be intentionally set wide to screen for this type of interference.

12.4. Also use a "Y" flag if the percent difference between the two columns is >40%. In such cases interference should be presumed even if not obvious and the analyte reporting limit should be raised to the higher value.

12.5. If the analyte is present on both columns within the established retention time values, and the RPD between the two values is  $\leq 40\%$ , the higher of the value is reported.

12.6. If a confirmed analyte concentration is less than the reporting limit, but above MDL level, the value may be "J" flagged at the PMs request. If the response of any analyte of interest exceeds



the calibration range of the system, dilute the extract so the analyte of interest falls within the range of the calibration curve and re-analyze.

12.7. After the data has been evaluated, the values are entered into the LIMS system which then processes the numbers for final reporting by using the following calculation:

<b>Concentration (ppm) = <math>\frac{(C \times FV)}{AmEx \times Dwt} \times DF</math></b>
where:
C = On-column concentration of product (□g/mL)
FV = Final volume of extract (mL)
AmEx= Amount of sample extracted (mL if water, g if soil)
Dwt = % total solids of soil sample, ignore if the sample is a wate
DF = Dilution factor

12.7.1. The Retention Time (RT) windows are defaulted to 0.05 minutes as discussed in Section 11.9.

## 12.8. Sample Quantitation

### 12.8.1. Water

<b>Concentration (µg/L) = <math>\frac{(Ax)(Is)(Vt)(DF)}{(Ais)(RRF)(Vo)(Vi)}</math></b>
where:
A <sub>X</sub> = Area of the quantitation peak for the compound being measured
I <sub>s</sub> = Amount of internal standard injected (ng).
V <sub>t</sub> = Volume of total extract
A <sub>IS</sub> = Peak area of the internal standard
RRF = Relative response factor for compound being measured
C <sub>X</sub> = Concentration of the analyte
V <sub>o</sub> = Volume of water extracted (ml)
V <sub>i</sub> = Volume of extract injected (µL)
DF = Sample Dilution Factor (1 for undiluted samples)

12.8.2. Sediment/Soil/Sludge (on a dry weight basis) and Waste (normally on a wet weight basis).

<b>Concentration (µg/kg) = <math>\frac{(Ax)(Is)(Vt)(DF)}{(Ais)(RRF)(Vi)(Ws)(D)}</math></b>
where:
A <sub>X</sub> = Area of the quantitation peak for the compound being measured
I <sub>s</sub> = Amount of internal standard injected (ng).
V <sub>t</sub> = Volume of total extract



DF = Sample Dilution Factor (1 for undiluted samples)
$A_{IS}$ = Peak area of the internal standard
RRF = Relative response factor for compound being measured
$V_i$ = Volume of extract injected ( $\mu$ L)
$W_s$ = Weight of sample extracted or diluted in grams
D = % Dry weight of sample = 1.0 on an as received basis

12.9. ARI will report the higher of the results as per Method 8000B. This approach is conservative relative to protection of the environment. In certain instances or for project specific requirements, it may be appropriate to report the lower result as per Method 8000C. This approach is only valid under the discretion of an experienced analyst and must be noted in the analyst's notes and in the project narrative.

### 13. Method Performance

13.1. The QA department measures method performance using a combination of annual method detection limit (MDL) studies, performance evaluation samples, and the monitoring of surrogate and spike recoveries.

13.2. Detection limits- detection limits for all analytes quantitated using this SOP are set using the low point of the initial calibration curve and validated by method detection limit studies.

13.2.1. MDL studies are performed each year for each analyte by each preparatory and analytical method.

13.2.2. MDL and reporting limit (RL) values may be found for each analyte in the ARI QAP

13.3. Laboratory precision and bias measurements are performed by monitoring surrogate and spike recoveries in samples and quality control samples.

13.3.1. Control limits are calculated from these recoveries.

13.4. These control limits are disseminated to the bench chemists and LIMS administrator for use in monitoring method performance in real time.

13.5. As these limits are updated regularly, their dynamic nature prevents their inclusion in this SOP. However, they may be found in the ARI QAP.

13.6. This method should be performed only by experienced GC analysts, or under the close supervision of such analysts.

13.7. The experience of the analyst must weigh heavily in the interpretation of the chromatogram. In the case of multi-component analytes such as Aroclors the analyst should rely primarily on pattern recognition.

13.8. QA maintains control charts for the recovery of surrogate standards and spiked compounds.



13.9. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

#### **14. Pollution Prevention**

14.1. Disposed expired standards into the designated barrel in the hazardous waste room.

14.2. Samples that are designated as hazardous waste by the LIMS “Hazardous Report” must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

#### **15. Data Assessment and Acceptance Criteria for Quality Control Measures**

15.1. Requirements relating to initial and continuing calibration are detailed in Section 11.0 of this document.

15.2. Method Blanks- The method blank must contain less than 1/2 the reporting limit of the targeted analytes.

##### **15.3. Surrogate Recoveries**

15.3.1. All method blanks, laboratory control samples, matrix spikes, matrix spike duplicates, duplicates, SRMs or other samples must have acceptable surrogate recoveries. Surrogate recoveries are considered unacceptable when they fall outside ARI’s statistically generated control limits found in the QAPP.

15.3.2. Surrogate recovery acceptance windows are ideally determined statistically from method and matrix specific laboratory data updated on a periodic basis. Certain methods or clients may specify project specific surrogate recovery acceptance windows.

15.3.3. These requirements do not apply to subsequent dilutions of samples where a prior analysis of the diluted sample extract shows acceptable surrogate recovery.

15.3.4. When mandated by contract-specific requirements, corrective actions must be performed in response to failure to meet project specific surrogate acceptance criteria, even when the criteria are labeled as advisory in the reference method.

15.3.5. Surrogate acceptance criteria are both matrix and concentration level specific (e.g. low level vs. medium level soils). When analyzing matrices or concentration levels for which no acceptance criteria are available, the closest approximation of available acceptance criteria may be provided as estimates for advisory purposes only.

##### **15.4. Laboratory Control Samples (LCS)**

15.4.1. The RPD values between the spiked herbicides values in an LCS/LCSD should be  $\leq$  30%. If the RPD exceed 30%, the analyst should examine the surrogate recovery of the LCS/LCSD. Since the surrogate is a measure of extraction efficiency, if the RPD between the surrogate recoveries of the LCS/LCSD is  $\leq$  30% then no corrective action is necessary.



If the RPD also exceed 30%, re-extraction of all samples may be required with the following exception:

15.4.2. The LCS recovery values should fall within the specified recovery acceptance limits. If an LCSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.

15.4.3. LCS recovery acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Project or method specific limits may supersede laboratory acceptance criteria.

15.4.4. When mandated by contract-specific requirements, corrective actions must be performed in response to failure to meet spike acceptance criteria, even when the criteria are labeled as advisory in the reference method.

15.4.5. Spike acceptance criteria are both matrix and concentration level specific (e.g. low level vs. medium level soils). When analyzing matrices or concentration levels for which no acceptance criteria are available, the closest approximation of available acceptance criteria may be provided as estimates for advisory purposes only.

#### 15.5. Matrix Spike/Matrix Spike Duplicates (MS/MSD)

15.5.1. Matrix Spike/Matrix Spike Duplicate recovery values should fall within the specified recovery acceptance limits. If a MSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.

15.5.2. MS/MSD recovery and RPD acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Certain methods or clients may require project specific MS/MSD recovery and RPD acceptance windows.

15.5.3. The RPD between the spiked herbicide concentrations in the MS/MSD should be  $\leq$  30%. If the RPD exceed 30%, the analyst should examine the surrogate recovery of the MS/MSD. Since the surrogate is a measure of extraction efficiency, if the RPD between the surrogate recoveries of the MS/MSD is  $\leq$  30% then no corrective action is necessary. If the RPD also exceed 30%, re-extraction may be required.

#### 15.6. Holding Times

15.6.1. Extracts should be analyzed within the extract holding time (40 days from the date of extraction.)

15.6.2. In the event that re-extraction due to an out of control event requires that samples be re-extracted after their extraction holding time has elapsed (seven days for water and fourteen days for tissues/solids) the analyst should analyze and report both extraction sets, whenever practical, distinguishing between the initial extraction and re-extraction on all deliverables.



This will document that the samples were originally extracted within holding times and may allow for comparisons that will determine whether data quality was affected by the samples being analyzed out of holding.

15.6.3. If any extracts are analyzed after the 40 day extract holding time has elapsed, the analyst shall document this in the analytical notes accompanying the data so that it may be included in the narrative

## 16. Corrective Actions for Out of Control Events

### 16.1. Initial and Continuing Calibrations-

16.1.1. If the RSD for a herbicide exceeds 20%, examine the initial calibration. If the failure is deemed to be the result of an improperly prepared calibration standard, re-prepare the standard and reanalyze it. Reanalyzing or replacing a single standard must NOT be confused with the practice of discarding individual calibration results for specific target compounds in order to pick and choose a set of results that will meet the RSD or correlation criteria for the linear model. The practice of discarding individual calibration results is addressed as a fourth alternative option, and is very specific as to how a set of results are chosen to be discarded. If a standard is reanalyzed, or a new standard is analyzed, then ALL of the results from the original analysis of the standard in question must be discarded. Further, the practice of running additional standards at other concentrations and then picking only those results that meet the calibration acceptance criteria is EXPRESSLY PROHIBITED, since the analyst has generated data that demonstrate that the linear model does not apply to all of the data.

16.1.2. If the high RSD is not due to an improperly prepared calibration standard, perform instrument maintenance. This maintenance includes, but is not limited to: replacing the inlet liner and liner packing, cleaning the inlet liner, cleaning or replacing the inlet seal, cleaning or replacing the inlet body, replacing the split line, cleaning the split arm, clipping a length from the front of the columns or guard column, or replacing the column or guard column. Any maintenance is to be documented in the "maintenance" section of the GC run logbook for that instrument. Return to control is demonstrated by the successful analysis of an initial calibration.

16.2. Continuing Calibration- All CVSs must have an average %D within 15% of the true value.. If the standard does not meet this QC limit, all samples run after the last acceptable standard must be re-analyzed, unless the GC Supervisor and/or PM allow the variance.

16.2.1. If the % D of the CVS is greater than 15%, the CVS may still be usable if one of the following conditions are met:



16.2.1.1. If all samples are clean and the CVSs are >15% high, then the data may be reported. The fact that the CVSs are high indicates that if there were hits in the samples they would have been detected.

16.2.1.2. If the CVS is out for a range and the sample requires dilution, both the dilution and the original sample run should be reported. There is no need to have passing CVSs for a value that will already be flagged.

16.2.1.3. If the CVSs fail again after the instrument has been brought back into control and the samples rerun, it can be assumed that the samples are responsible for the CVS failure and no further corrective action is necessary. Note: To meet 18.2.1.3, both the original bracket and the reanalyzed bracket must be identical.

16.2.2. The analyst should evaluate the CVS problem and recheck any previous changes due to instrument maintenance, (i.e. leaks from any welds, position of the column inserted in the injector, jagged edges at column clips, etc). If corrective action brings the instrument back into compliance, then the run may continue from that point. All samples run since the last acceptable CVS must be rerun. Instrument maintenance may include some or all of the actions listed in Section 18.1.2. Changing the column or the inlet body requires a new initial calibration whether or not CVS meet the old calibration.

16.2.3. If the out-of-control event cannot be reconciled, a new initial calibration must be run and the samples contained in the unacceptable bracket re-analyzed.

### 16.3. Surrogates-

16.3.1. If the surrogate compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.3.2. If the surrogate recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the surrogate compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the surrogate compound recoveries meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of surrogate compound spiking solution was added either recalculate surrogate recoveries based on the actual amount of surrogate compound spiking solution added, or re-extract/reanalyze the sample, adding the correct amount of surrogate spiking solution. If the surrogate compound spiking solution and/or surrogate calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.3.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will mostly involve maintenance similar to the



maintenance discussed in Section 18.1.2. Verify that the surrogate recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.3.4. If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was matrix effect, take the following corrective action steps.

16.3.4.1. Re-extract and reanalyze the sample. EXCEPTION: If surrogate compound recoveries in a sample used for a matrix spike and/or matrix spike duplicate were considered unacceptable, then it should be re-extracted/reanalyzed only if the surrogate compound recoveries were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of unspiked duplicates of a given sample which is outside acceptance criteria.

16.3.4.2. If the surrogate compound recoveries meet acceptance criteria in the re-extracted/reanalyzed sample, then the problem with the initial analysis is deemed to be within the laboratory's control. Therefore, submit data only from the re-extraction/reanalysis if the re-extraction was performed within holding time, otherwise report both sets of data.

16.3.4.3. If the surrogate compound recoveries fail to meet the acceptance criteria in the re-extracted/reanalyzed sample, then submit data from both analyses, distinguishing between the initial analysis and the re-extraction/reanalysis on all deliverables.

16.3.4.4. Method Blanks- Corrective action for a method blank which fails acceptance criteria may involve re-extraction and reanalysis of all associated samples and/or "B" flagging of the associated sample data. Each occurrence will be evaluated on an individual basis upon consultation with the Project Manager, the client, the Laboratory Supervisor, and the Laboratory Manager.

#### 16.4. Laboratory Control Samples

16.4.1. If the LCS compounds fail to meet their recovery acceptance criteria, first reanalyze the extract.

16.4.2. If the LCS recoveries do not meet their recovery acceptance criteria after reanalysis, check calculations, sample preparation logs, the LCS compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the LCS compound recoveries and RPD's (if applicable) meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of LCS compound spiking solution was added either recalculate LCS recoveries based on the





actual amount of LCS compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of LCS spiking solution. If the LCS compound spiking solution and/or LCS calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.4.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the LCS recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.4.4. If the LCS compounds still fail to meet their acceptance criteria, re-extract and reanalyze the LCS and all associated samples and QC samples if deemed appropriate (i.e. after consideration of all batch QC data) or mandated by contract-specific requirements. Any decision to forgo re-extraction/reanalysis based on failure to meet LCS acceptance criteria will require approval of the Project Manager and the Lab Manager, at a minimum.

#### 16.5. Matrix Spike/Matrix Spike Duplicates

16.5.1. If the MS/MSD compounds fail to meet their acceptance criteria, reanalyze the extracts.

16.5.2. If this fails to bring the recoveries or RPDs into compliance, check calculations, sample preparation logs, the MS/MSD compound spiking and calibration solutions and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the MS/MSD compound recoveries and RPD's (if applicable) meet their acceptance criteria. If the sample preparation logs indicate that the incorrect amount of MS/MSD compound spiking solution was added either recalculate MS/MSD recoveries based on the actual amount of MS/MSD compound spiking solution added or re-extract/reanalyze the samples, adding the correct amount of MS/MSD spiking solution. If the MS/MSD compound spiking solution and/or MS/MSD calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples.

16.5.3. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Verify that the MS/MSD recoveries meet their acceptance criteria. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.5.4. If the MS/MSD compounds still fail to meet their acceptance criteria, an assessment of batch QC data is necessary in order to determine the possibility of a matrix effect. If the RPD's are within acceptance limits (particularly for non-native MS/MSD analytes) and all of the batch QC elements which are not affected by the sample matrix are in control (e.g. method blank, LCS, calibration checks) and there is no evidence of improper spiking levels, the poor recovery may be attributed to matrix effects. Additional consideration also needs to



be given to the ratio of spike to native analyte, as well as sample heterogeneity, as these factors may have an adverse effect on both spike recovery and RPD values. If matrix effect is determined to be the cause, the associated data must be flagged, but re-preparation and reanalysis is not required. Examination of spike recoveries in the matrix spike duplicate is also pertinent to assessing matrix effects.

16.5.5. If any of the batch QA elements which are not affected by the sample matrix are out of control, or there is evidence that spiking may have been improperly performed, the matrix spike and matrix spike duplicate must be re-extracted and reanalyzed. If insufficient sample is available or if the extraction holding time has elapsed, the laboratory must notify the client and flag the associated data and/or document the problem in the narrative.

16.6. Sample Dilution- Should the quantitated value of any analyte exceed the working range of the curve, a dilution must be performed such that the analyte's quantitated value is within the curve range.

16.6.1. Additional internal standard must be added to the diluted extract to maintain the required 20 ng/ $\mu$ L of each internal standard in the extracted volume.

16.6.2. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

16.6.3. When a herbicide peak saturates the detector the analyst must do one of the following:

16.6.3.1. Flag all affected analytes with an S flag.

16.6.3.2. Analyze an Instrument Blank consisting of clean solvent until the system has been decontaminated.

## **17. Contingencies for Handling Out-of-Control or Unacceptable Data**

17.1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction using a 'Request for Re-extraction/Re-analysis Form (Form 0030F). Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

17.2. See Sections 18.1-18.2 for guidance on dealing with initial and continuing calibration out-of-control events.

17.3. See Section 18.3 for guidance on dealing with internal standard out-of-control events.

17.4. See Section 18.4 for guidance on dealing with surrogate out-of-control events.

17.5. See Section 18.5 for guidance on dealing with method blank related out-of-control events.

17.6. See Section 18.6 for guidance on dealing with laboratory control sample related out-of-control events.

17.7. See Section 18.7 for guidance on dealing with MS/MSD related out-of-control events.



## **18. Waste Management**

- 18.1. All extract vials must be disposed of by placing them in the blue hazardous waste drum in the lab set aside for this purpose. No vials may be thrown in the trash or receptacles not expressly designated for this purpose.
- 18.2. All solvents must be disposed of by pouring them out over charcoal. No solvent may be poured down the drain or disposed of in any other non-hygienic manner.
- 18.3. All spent charcoal must be disposed of by placing it in the charcoal disposal bin located in the extractions lab.

## **19. Method References**

- 19.1. SW-846,

**APPENDIX B**

**LABORATORY CERTIFICATIONS**

# Scope of Accreditation

## Analytical Resources, Incorporated

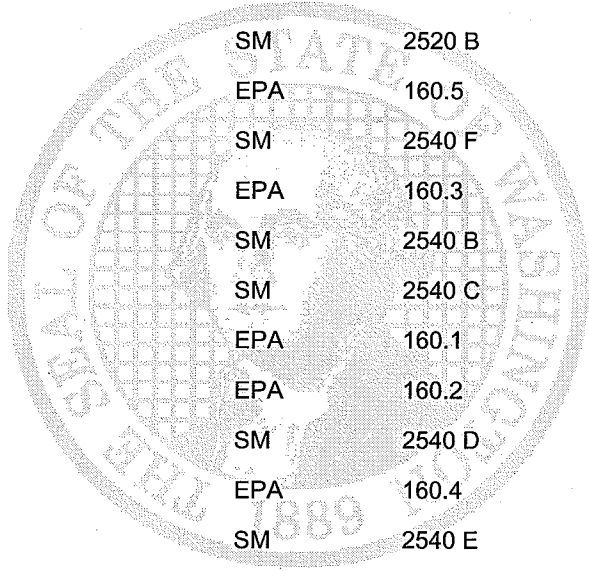
Tukwila, WA

is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation may apply to any of the following matrix types: non-potable water, drinking water, solid and chemical materials, and air and emissions. Accreditation for all parameters is final unless indicated otherwise in a note. Accreditation is for the latest version of a method unless otherwise specified in a note. EPA refers to the U.S. Environmental Protection Agency. SM refers to American Public Health Association's publication, Standard Methods for the Examination of Water and Wastewater, 18th, 19th or 20th Edition, unless otherwise noted. ASTM stands for the American Society for Testing and Materials. PSEP stands for Puget Sound Estuary Program. Other references are detailed in the notes section.

Matrix Type/Parameter Name	Reference	Method Number	Notes
<b>Drinking Water</b>			
Arsenic	EPA	200.8	1
Arsenic	EPA	200.9	1
Copper	EPA	200.8	1
Lead	EPA	200.9	1
Lead	EPA	200.8	1
Purgeable Organic Compounds	EPA	524.2	1
Regulated VOCs	EPA	524.2	1
Trihalomethanes	EPA	524.2	8
Unregulated VOCs	EPA	524.2	1
Vinyl Chloride	EPA	524.2	1
<b>Non-potable Water</b>			
Acidity	EPA	305.1	1
Acidity	SM	2310 B(4c)	1
Alkalinity, Total	EPA	310.1	
Alkalinity, Total	SM	2320 B(4c)	
Ammonia	EPA	350.3	1
Ammonia	SM 19/20	4500-NH3 D	1
Ammonia	SM 19/20	4500-NH3 G	1
Ammonia	EPA	350.1	1

<b>Matrix Type/Parameter Name</b>	<b>Reference</b>	<b>Method Number</b>	<b>Notes</b>
Biochemical Oxygen Demand, BOD/CBOD	EPA	405.1	1
Biochemical Oxygen Demand, BOD/CBOD	SM	5210 B	1
Bromide	SM	4110 B	1
Bromide	EPA	300.0	1
Chemical Oxygen Demand (COD)	SM	5220 D	1
Chemical Oxygen Demand (COD)	EPA	410.4(7.3)	1
Chloride	EPA	300.0	1
Chloride	SM	4500-Cl- E	1,8
Chloride	EPA	325.2	1,8
Chloride	SM	4110 B	1
Chromium, Hexavalent	SM	3500-Cr D	1
Color	EPA	110.2	1
Color	SM	2120 B	1
Cyanide, Total	EPA	335.2(8.10)	1,8
Cyanide, Weak Acid Dissociable	SM	4500-CN J	
Cyanides, Amenable to Chlorination	SM	4500-CN G	1
Cyanides, Amenable to Chlorination	EPA	335.1	1
Dissolved Oxygen	SM	4500-O C	1
Dissolved Oxygen	EPA	360.2	1
Fluoride	EPA	300.0	1
Fluoride	SM	4110 B	1
Fluoride	EPA	340.2	1
Hardness, Total (as CaCO <sub>3</sub> )	SM	2340 B	1
Hexane Extractable Material	EPA	1664	1
Nitrate	EPA	353.2	1
Nitrate	SM	4500-NO3 F	1
Nitrate	SM	4110 B	1
Nitrate	EPA	300.0	1
Nitrate + Nitrite	EPA	353.2	1
Nitrate + Nitrite	SM	4500-NO3 F	1

Matrix Type/Parameter Name	Reference	Method Number	Notes
Nitrite	EPA	300.0	1
Nitrite	EPA	353.2	1
Nitrite	SM	4110 B	1
Nitrogen, Total Kjeldahl	EPA	351.4	1
Orthophosphate	EPA	300.0	1
Orthophosphate	SM	4110 B	1
Orthophosphate	EPA	365.2	
pH	EPA	150.1	1
Phenolics, Total Recoverable	EPA	420.1	1
Phosphorus, Total	EPA	365.2	1
Phosphorus, Total	SM	4500-P E	
Salinity	SM	2520 B	
Solids, Settleable	EPA	160.5	1
Solids, Settleable	SM	2540 F	1
Solids, Total	EPA	160.3	1
Solids, Total	SM	2540 B	1
Solids, Total Dissolved	SM	2540 C	1
Solids, Total Dissolved	EPA	160.1	1
Solids, Total Suspended	EPA	160.2	1
Solids, Total Suspended	SM	2540 D	1
Solids, Total Volatile	EPA	160.4	1
Solids, Total Volatile	SM	2540 E	
Specific Conductance	EPA	120.1	1
Specific Conductance	SM	2510 B	1
Sulfate	EPA	300.0	1
Sulfate	EPA	375.2	1
Sulfate	SM	4110 B	1
Sulfide	SM	4500-S2 D	1
Sulfide	SM	4500-S2 E	
Sulfide	EPA	376.1	



Matrix Type/Parameter Name	Reference	Method Number	Notes
Sulfide	EPA	376.2	1
Sulfite	EPA	377.1	1
Total Organic Carbon	EPA	415.1	1
Total Organic Carbon	SM	5310 B	1
Turbidity	EPA	180.1	1
Turbidity	SM	2130 B	1
Aluminum	EPA	200.7	1
Aluminum	EPA	200.8	
Antimony	EPA	200.8	
Antimony	EPA	200.9	
Antimony	EPA	200.7	1
Antimony	EPA	204.2	1
Arsenic	EPA	200.7	1
Arsenic	EPA	200.8	
Arsenic	EPA	200.9	
Arsenic	EPA	206.2	1
Barium	EPA	200.7	1
Barium	EPA	200.8	
Beryllium	EPA	200.7	1
Beryllium	EPA	200.8	
Boron	EPA	200.7	1
Cadmium	EPA	200.9	
Cadmium	EPA	213.2	1
Cadmium	EPA	200.7	1
Cadmium	EPA	200.8	
Calcium	EPA	200.8	
Calcium	EPA	200.7	1
Chromium	EPA	200.7	1
Chromium	EPA	200.8	
Cobalt	EPA	200.8	



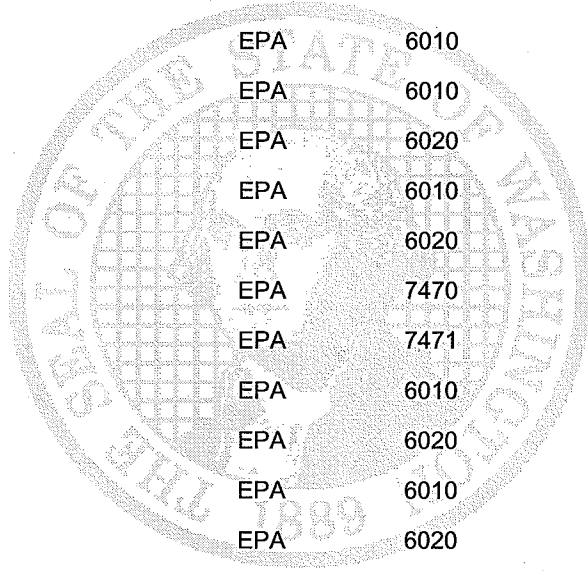


Matrix Type/Parameter Name	Reference	Method Number	Notes
Cobalt	EPA	200.7	1
Copper	EPA	200.7	1
Copper	EPA	200.8	
Iron	EPA	200.7	1
Iron	EPA	200.8	
Lead	EPA	200.7	1
Lead	EPA	200.8	
Lead	EPA	239.2	1
Lead	EPA	200.9	
Magnesium	EPA	200.8	
Magnesium	EPA	200.7	1
Manganese	EPA	200.8	
Manganese	EPA	200.7	1
Mercury	EPA	245.1	1
Molybdenum	EPA	200.8	
Molybdenum	EPA	200.7	1
Nickel	EPA	200.8	
Nickel	EPA	200.7	1
Potassium	EPA	200.7	1
Potassium	EPA	200.8	
Selenium	EPA	200.7	1
Selenium	EPA	200.9	
Selenium	EPA	200.8	
Selenium	EPA	270.2	1
Silica	SM	3120 B	
Silica	EPA	200.7	1
Silver	EPA	200.7	1
Silver	EPA	200.9	
Silver	EPA	200.8	
Silver	EPA	272.2	1

Matrix Type/Parameter Name	Reference	Method Number	Notes
Sodium	EPA	200.7	1
Sodium	EPA	200.8	
Strontium	EPA	200.7	1
Thallium	EPA	279.2	1
Thallium	EPA	200.9	
Thallium	EPA	200.7	1
Thallium	EPA	200.8	
Tin	EPA	200.7	1
Titanium	EPA	200.7	1
Uranium	EPA	200.8	
Vanadium	EPA	200.8	
Vanadium	EPA	200.7	1
Zinc	EPA	200.8	
Zinc	EPA	200.7	1
VOA & Semi-VOA Compounds	ARI SOP	427S	4
Butyl-tin species	ARI	802S	5
Vinyl Chloride/1,1-DCE	ARI	703S	2,6
Fecal Coliform - count	SM	9222 D	
Total Coli - count MF	SM	9222 B2,5,6	
<b>Solid and Chemical Materials</b>			
Bromide	EPA	9056	1
Chloride	EPA	9251	1
Chloride	EPA	9056	1
Chromium, Hexavalent	EPA	7196	1
Cyanide, Free	EPA	9014(7.2)	1
Cyanide, Total	EPA	9010	1
Cyanide, Total	EPA	9014(7.2)	1
Cyanides, Amenable to Chlorination	EPA	9010	1
Fluoride	EPA	9214	1
Fluoride	EPA	9056	1

Matrix Type/Parameter Name	Reference	Method Number	Notes
Hexane Extractable Material	EPA	9071 B	1
Nitrate	EPA	9056	1
Nitrite	EPA	9056	1
Orthophosphate	EPA	9056	1
pH	EPA	9040	
pH (non-aqueous)	EPA	9045	1
Phenolics, Total Recoverable	EPA	9065	1
Sulfate	EPA	9056	1
Sulfate	EPA	9036	
Sulfide	EPA	9030	1
Sulfide	EPA	9034	
Total Organic Carbon	EPA	9060	
Total Organic Carbon (Sed)	PSEP	1986	8
Aluminum	EPA	6020	
Aluminum	EPA	6010	1
Antimony	EPA	7041	1
Antimony	EPA	6020	
Antimony	EPA	6010	1
Arsenic	EPA	6010	1
Arsenic	EPA	6020	
Arsenic	EPA	7060	1
Barium	EPA	6010	1
Barium	EPA	6020	
Beryllium	EPA	6010	1
Beryllium	EPA	6020	
Boron	EPA	6010	1
Cadmium	EPA	6010	1
Cadmium	EPA	6020	
Cadmium	EPA	7131	1
Calcium	EPA	6020	

Matrix Type/Parameter Name	Reference	Method Number	Notes
Calcium	EPA	6010	1
Chromium	EPA	6010	1
Chromium	EPA	6020	
Cobalt	EPA	6010	1
Cobalt	EPA	6020	
Copper	EPA	6010	1
Copper	EPA	6020	
Iron	EPA	6020	
Iron	EPA	6010	1
Lead	EPA	7421	1
Lead	EPA	6020	
Lead	EPA	6010	1
Magnesium	EPA	6010	1
Magnesium	EPA	6020	
Manganese	EPA	6010	1
Manganese	EPA	6020	
Mercury, Liquid Waste	EPA	7470	1
Mercury, Solid Waste	EPA	7471	1
Molybdenum	EPA	6010	1
Molybdenum	EPA	6020	
Nickel	EPA	6010	1
Nickel	EPA	6020	
Potassium	EPA	6010	1
Potassium	EPA	6020	
Selenium	EPA	6020	
Selenium	EPA	7740	1
Selenium	EPA	6010	1
Silica	EPA	6020	
Silica	EPA	6010	1
Silver	EPA	6010	1



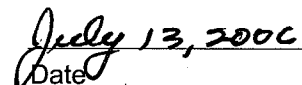
Matrix Type/Parameter Name	Reference	Method Number	Notes
Silver	EPA	6020	
Silver	EPA	7761	1
Sodium	EPA	6020	
Sodium	EPA	6010	1
Strontium	EPA	6010	1
Thallium	EPA	6020	
Thallium	EPA	7841	1
Thallium	EPA	6010	1
Tin	EPA	6010	1
Titanium	EPA	6010	1
Uranium	EPA	6020 Mod	
Vanadium	EPA	6010	1
Vanadium	EPA	6020	
Zinc	EPA	6010	1
Zinc	EPA	6020	
Chlorinated Herbicides	EPA	8151	1
Organochlorine Pesticides	EPA	8081	1
Petroleum Hydrocarbons, Extractable	WDOE	EPH	1
Petroleum Hydrocarbons, Volatile	WDOE	VPH	1
Phenols	EPA	8041	
Polychlorinated Biphenyls	EPA	8082	1,7
Total Pet Hydrocarbons - Diesel	WDOE	NWTPH-Dx	1,3
Total Pet Hydrocarbons - Gasoline	WDOE	NWTPH-Gx	1,3
Volatile Aromatics	EPA	8021	1
BNA Extr (Semivolatile) Organics	EPA	8270	1
Polycyclic Aromatic HC (PAHs)	EPA	8270 SIM	1,2
Volatile Organic Compounds	EPA	8260	1
Particle Size Distribution	ASTM	D 422	
Particle Size Distribution (Sed)	PSEP	1986	

**Matrix Type/Parameter Name**                      **Reference**    **Method Number**    **Notes**

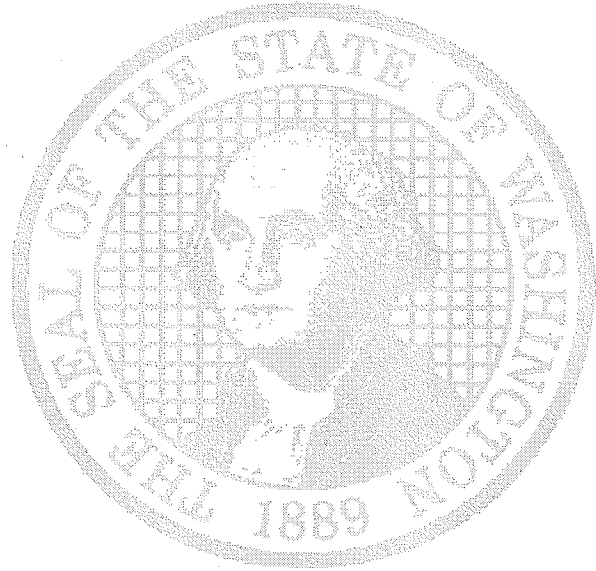
**Accredited Parameter Note Detail**

(1) Recognition of Oregon NELAP accreditation. (2) GC-MS Selective Ion Monitoring (SIM). (3) Washington Department of Ecology Analytical Methods for Petroleum Hydrocarbons, Publication Number ECY 97-602, June 1997. (4) ARI SOP for "Water Soluble Non-halogenated Volatile and Semivolatile Organic Compounds," including glycols. (5) ARI SOP, a modification of Krone Procedure. (6) ARI SOP for Vinyl Chloride determination. (7) Includes capability for low levels in aqueous samples using a modified hexane extraction. (8) Provisional pending an acceptable proficiency testing (PT) result (WAC 173-50-110).

  
\_\_\_\_\_  
Authentication Signature

  
\_\_\_\_\_  
Date

Stewart M. Lombard, Lab Accreditation Unit Supervisor



## Parameters Denied Accreditation

### Analytical Resources, Incorporated

Tukwila, WA

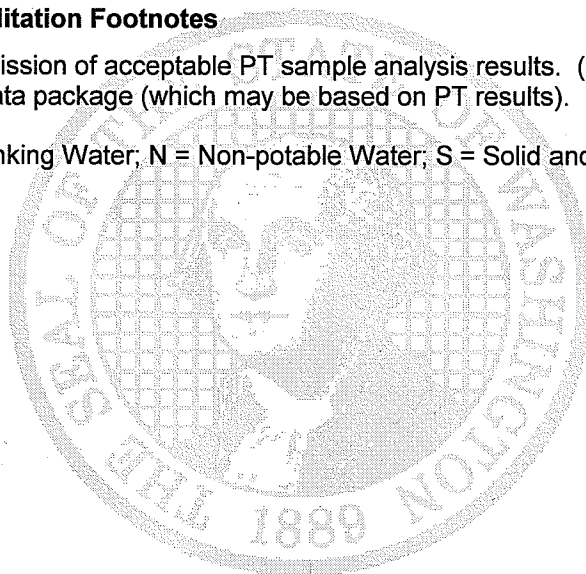
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Parameter Name	Reference	Method Number	Notes	Matrix
Enterococcus/Fecal Strep	SM	9230 C2ab,3,4,5	2	N
Heterotrophic Plate Count	SM	9215 D	2	N
Strontium	EPA	200.8	1	N
Strontium	EPA	6020	1	S
Tin	EPA	200.8	1	N
Titanium	EPA	200.8	1	N
Titanium	EPA	6020	1	S

#### Denied Parameter Accreditation Footnotes

(1) Withheld pending submission of acceptable PT sample analysis results. (2) Withheld pending acceptable lab SOP and data package (which may be based on PT results).

Matrix Definitions - D = Drinking Water, N = Non-potable Water, S = Solid and Chemical Material; A = Air and Emissions.





# Oregon

## Environmental Laboratory Accreditation Program



Department of Agriculture, Laboratory Division  
 Department of Environmental Quality, Laboratory Division  
 Department of Human Services, Public Health Laboratory

Public Health Laboratory  
 1717 SW 10th Avenue  
 Portland, OR 97201  
 (503) 229-5505  
 FAX (503) 229-5682  
 TTY (503) 731-4031

NELAP Recognized

### ORELAP Fields of Accreditation

ORELAPID: WA 100006

EPA Code:

### Analytical Resources Inc.

4611 S. 134th Place  
 Tukwila, WA, 98168

Certificate: WA 100006-00

Issue Date: 5/13/2006 2:12:2. Expiration Date: 5/11/2007 8:21:5

As of 5/13/2006 2:12:27 PM this list supercedes all previous lists for this certificate number.  
 Customers: Please verify the current accreditation standing with ORELAP.

### MATRIX: Drinking Water

<u>Reference</u>	<u>Code</u>	<u>Description</u>
EPA 200.8	10014809	ICP/MS - metals
<u>Analyte Code</u>	<u>Analyte</u>	
1000	Aluminum	
1005	Antimony	
1010	Arsenic	
1015	Barium	
1020	Beryllium	
1030	Cadmium	
1040	Chromium	
1050	Cobalt	
1055	Copper	
1070	Iron	
1075	Lead	
1100	Molybdenum	
1105	Nickel	
1140	Selenium	
1150	Silver	
1165	Thallium	
1170	Thorium	
3035	Uranium	
1185	Vanadium	
EPA 200.9	10015608	Metals by Graphite Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1005	Antimony	
1010	Arsenic	
1030	Cadmium	
1075	Lead	
1140	Selenium	
1150	Silver	
1165	Thallium	
EPA 524.2	10088809	Volatile Organic Compounds GC/MS Capillary Column
<u>Analyte Code</u>	<u>Analyte</u>	
5110	1,1,2,2-Tetrachloroethane	
5165	1,1,2-Trichloroethane	
4630	1,1-Dichloroethane	
4640	1,1-Dichloroethylene	
4670	1,1-Dichloropropene	



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5150	1,2,3-Trichlorobenzene
5180	1,2,3-Trichloropropane
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
4615	1,3-Dichlorobenzene
4660	1,3-Dichloropropane
4620	1,4-Dichlorobenzene
4535	2-Chlorotoluene
4540	4-Chlorotoluene
5870	4-Isopropyltoluene
4375	Benzene
4385	Bromobenzene
4390	Bromochloromethane
4950	Bromomethane (Methyl bromide)
4455	Carbon tetrachloride
4475	Chlorobenzene
4485	Chloroethane
105	Chloromethane
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4595	Dibromomethane
4625	Dichlorodifluoromethane
4650	Dichloromethane (DCM; Methylene chloride)
4765	Ethylbenzene
4835	Hexachlorobutadiene
4900	Isopropylbenzene
5240	m+p-xylene
5005	Naphthalene
5090	n-Propylbenzene
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane
5235	Vinyl chloride

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<b>MATRIX: Non-Potable Water</b>		
<b>Reference</b>	<b>Code</b>	<b>Description</b>
EPA 110.1	10005206	Color - Colorimetric, ADMI @ pH 7.6
<u>Analyte Code</u>	<u>Analyte</u>	
1605	Color	
EPA 120.1	10006403	Conductance - Specific @ 25 C
<u>Analyte Code</u>	<u>Analyte</u>	
1610	Conductivity	
EPA 1311	10118806	Toxicity Characteristic Leaching Procedure
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 1312	10119003	Synthetic Precipitation Leaching Procedure
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 150.1	10008409	pH - Electrometric Measurement
<u>Analyte Code</u>	<u>Analyte</u>	
1900	pH	
EPA 160.1	10009208	Total Dissolved Solids, dried @ 180 C
<u>Analyte Code</u>	<u>Analyte</u>	
1955	Residue-filterable (TDS)	
EPA 160.2	10009606	Total Suspended Solids, 0.2um dried @105C
<u>Analyte Code</u>	<u>Analyte</u>	
1960	Residue-nonfilterable (TSS)	
EPA 160.3	10010001	Total Solids, dried @ 103-105 C.
<u>Analyte Code</u>	<u>Analyte</u>	
1950	Residue-total	
EPA 160.4	10010409	Total Volatile Solids, ignition @ 550 C.
<u>Analyte Code</u>	<u>Analyte</u>	
1970	Residue-volatile	
EPA 160.5	10010807	Settleable solids
<u>Analyte Code</u>	<u>Analyte</u>	
1965	Residue-settleable	
EPA 1664A	10127807	N-Hexane Extractable Material
<u>Analyte Code</u>	<u>Analyte</u>	
109	Hexane Extractable Material	
EPA 180.1	10011800	Turbidity - Nephelometric
<u>Analyte Code</u>	<u>Analyte</u>	
2055	Turbidity	
EPA 200.7	10014003	ICP - metals
<u>Analyte Code</u>	<u>Analyte</u>	
1000	Aluminum	
1005	Antimony	
1010	Arsenic	
1015	Barium	
1020	Beryllium	
1025	Boron	
1030	Cadmium	
1035	Calcium	
1040	Chromium	
1050	Cobalt	
1055	Copper	

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1070	Iron		
1075	Lead		
1085	Magnesium		
1090	Manganese		
1100	Molybdenum		
1105	Nickel		
1125	Potassium		
1140	Selenium		
1145	Silicon		
1150	Silver		
1155	Sodium		
1160	Strontium		
1165	Thallium		
1175	Tin		
1180	Titanium		
1185	Vanadium		
1190	Zinc		
<hr/>			
EPA 204.2		10017808	Antimony by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1005	Antimony		
<hr/>			
EPA 206.2		10018607	Arsenic by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1010	Arsenic		
<hr/>			
EPA 213.2		10024007	Cadmium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1030	Cadmium		
<hr/>			
EPA 239.2		10034205	Lead by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1075	Lead		
<hr/>			
EPA 245.1		10036609	Mercury by Cold Vapor Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1095	Mercury		
<hr/>			
EPA 270.2		10045406	Selenium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1140	Selenium		
<hr/>			
EPA 272.2		10046807	Silver by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1150	Silver		
<hr/>			
EPA 279.2		10048803	Thallium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>		
1165	Thallium		
<hr/>			
EPA 300.0		10053200	Inorganic Anions in water by Ion Chromatography
<u>Analyte Code</u>	<u>Analyte</u>		
1540	Bromide		
1575	Chloride		
1730	Fluoride		
1810	Nitrate as N		
1840	Nitrite as N		
1870	Orthophosphate as P		
2000	Sulfate		

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EPA 3005A	10133207	Acid Digestion of waters for Total Recoverable or Dissolved Metals
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3010A	10133605	Acid Digestion of Aqueous samples and Extracts for Total Metals
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3020A	10134404	Acid Digestion of Aqueous samples and Extracts for Total Metals for Anal
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 305.1	10054203	Acidity using H2O2 procedure
<u>Analyte Code</u>	<u>Analyte</u>	
1500	Acidity, as CaCO3	
EPA 325.2	10057202	Chloride - Colorimetric, automated Ferricyanide, II
<u>Analyte Code</u>	<u>Analyte</u>	
1575	Chloride	
EPA 335.1	10060001	Cyanides, amenable to chlorination
<u>Analyte Code</u>	<u>Analyte</u>	
1510	Amenable cyanide	
EPA 335.2	10060409	Cyanide - Titrimetric; Spectrophotometric
<u>Analyte Code</u>	<u>Analyte</u>	
1635	Cyanide	
EPA 340.2	10062201	Fluoride - Ion selective electrode
<u>Analyte Code</u>	<u>Analyte</u>	
1730	Fluoride	
EPA 350.1	10063602	Ammonia Nitrogen - Colorimetric, Auto Phenate
<u>Analyte Code</u>	<u>Analyte</u>	
1515	Ammonia as N	
EPA 350.3	10064401	Ammonia Nitrogen - Ion Selective Electrode
<u>Analyte Code</u>	<u>Analyte</u>	
1515	Ammonia as N	
EPA 351.4	10066203	Kjeldahl Nitrogen - Ion Selective Electrode
<u>Analyte Code</u>	<u>Analyte</u>	
1795	Kjeldahl nitrogen - total	
EPA 3510C	10138202	Separatory Funnel Liquid-liquid extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3520C	10139001	Continuous Liquid-liquid extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 353.2	10067604	Nitrate/Nitrite Nitrogen - Automated, Cadmium
<u>Analyte Code</u>	<u>Analyte</u>	
1820	Nitrate+nitrite	
EPA 3550C	10142004	Ultrasonic Extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 360.2	10069406	Dissolved Oxygen - Winkler
<u>Analyte Code</u>	<u>Analyte</u>	
1880	Oxygen, dissolved	
EPA 3630C	10146802	Silica gel cleanup
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	

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<b>EPA 3640A</b>		<b>10147203</b>	<b>Gel Preparation Cleanup</b>
<u>Analyte Code</u>	<u>Analyte</u>		
125	Extraction/Preparation		
<b>EPA 365.2</b>		<b>10070403</b>	<b>Phosphorous - Colorimetric, single reagent</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1910	Phosphorus, total		
<b>EPA 3660B</b>		<b>10148400</b>	<b>Sulfur cleanup</b>
<u>Analyte Code</u>	<u>Analyte</u>		
125	Extraction/Preparation		
<b>EPA 375.2</b>		<b>10073004</b>	<b>Sulfate - Colorimetric, Automated, Methylthymol.</b>
<u>Analyte Code</u>	<u>Analyte</u>		
2000	Sulfate		
<b>EPA 376.2</b>		<b>10074609</b>	<b>Sulfide - Colorimetric, Methylene Blue.</b>
<u>Analyte Code</u>	<u>Analyte</u>		
2005	Sulfide		
<b>EPA 377.1</b>		<b>10075000</b>	<b>Sulfite - Titrimetric.</b>
<u>Analyte Code</u>	<u>Analyte</u>		
2015	Sulfite-SO3		
<b>EPA 405.1</b>		<b>10075602</b>	<b>Biochemical Oxygen Demand (5 days @ 20 C).</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1530	Biochemical oxygen demand		
<b>EPA 410.4</b>		<b>10077404</b>	<b>Chemical Oxygen Demand - Colorimetric, Automated.</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1565	Chemical oxygen demand		
<b>EPA 415.1</b>		<b>10078407</b>	<b>Organic carbon - Combustion or Oxidation</b>
<u>Analyte Code</u>	<u>Analyte</u>		
2040	Total Organic Carbon		
<b>EPA 420.1</b>		<b>10079400</b>	<b>Phenolics - Spectrophotometric, manual.</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1905	Total Phenolics		
<b>EPA 5000</b>		<b>10152600</b>	<b>Sample Preparation for Volatile Organics</b>
<u>Analyte Code</u>	<u>Analyte</u>		
125	Extraction/Preparation		
<b>EPA 5030B</b>		<b>10153409</b>	<b>Purge and trap for aqueous samples</b>
<u>Analyte Code</u>	<u>Analyte</u>		
125	Extraction/Preparation		
<b>EPA 6010B</b>		<b>10155609</b>	<b>ICP - AES</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1000	Aluminum		
1005	Antimony		
1010	Arsenic		
1015	Barium		
1020	Beryllium		
1025	Boron		
1030	Cadmium		
1035	Calcium		
1040	Chromium		
1050	Cobalt		
1055	Copper		
1070	Iron		
1075	Lead		



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EPA 7196A	10162400	Chromium Hexavalent colorimetric
<u>Analyte Code</u>	<u>Analyte</u>	
1045	Chromium VI	
EPA 7421	10164600	Lead by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1075	Lead	
EPA 7470A	10165807	Mercury in Liquid Waste by by Cold Vapor Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1095	Mercury	
EPA 7740	10168602	Selenium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1140	Selenium	
EPA 7761	10169809	Silver by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1150	Silver	
EPA 7841	10170602	Thallium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1165	Thallium	
EPA 8000B	10172200	Determinative Chromatographic Separations
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 8021B	10174808	Aromatic and Halogenated Volatiles by GC with PID and/or ECD Purge &
<u>Analyte Code</u>	<u>Analyte</u>	
5240	m+p-xylene	
5000	Methyl tert-butyl ether (MTBE)	
5250	o-Xylene	
EPA 8081B	10178800	Organochlorine Pesticides by GC/ECD
<u>Analyte Code</u>	<u>Analyte</u>	
8580	2,4'-DDD	
8585	2,4'-DDE	
8590	2,4'-DDT	
7355	4,4'-DDD	
7360	4,4'-DDE	
7365	4,4'-DDT	
7025	Aldrin	
7110	alpha-BHC (alpha-Hexachlorocyclohexane)	
7240	alpha-Chlordane	
7115	beta-BHC (beta-Hexachlorocyclohexane)	
7250	Chlordane (tech.)	
7300	Chlorpyrifos	
7925	cis-Nonachlor	
8550	Dacthal (DCPA)	
7105	delta-BHC	
7470	Dieldrin	
7510	Endosulfan I	
7515	Endosulfan II	
7520	Endosulfan sulfate	
7540	Endrin	
7530	Endrin aldehyde	
7535	Endrin ketone	
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	

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7685	Heptachlor
7690	Heptachlor epoxide
7735	Kelthane (Diclofol)
7810	Methoxychlor
7825	Methyl parathion (Parathion, methyl)
7870	Mirex
274	Oxadiazon
3890	Oxychlorane
7950	Parathion
7955	Parathion, ethyl
8250	Toxaphene (Chlorinated camphene)
7910	trans Nanochlor
8295	Trifluralin (Treflan)

**EPA 8082 10179007 Polychlorinated Biphenyls (PCBs) by GC/ECD**

<u>Analyte Code</u>	<u>Analyte</u>
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
184	Aroclor-1262 (PCB 1262)
103	Aroclor-1268 (PCB-1268)

**EPA 8151A 10183207 Chlorinated Herbicides by GC/ECD**

<u>Analyte Code</u>	<u>Analyte</u>
8655	2,4,5-T
8545	2,4-D
8560	2,4-DB
8555	Dalapon
8595	Dicamba
8605	Dichloroprop (Dichlorprop)
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)
7775	MCPA
7780	MCPP
8650	Silvex (2,4,5-TP)

**EPA 8260B 10184802 Volatile Organic Compounds by purge and trap GC/MS**

<u>Analyte Code</u>	<u>Analyte</u>
5105	1,1,1,2-Tetrachloroethane
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
4670	1,1-Dichloropropene
5150	1,2,3-Trichlorobenzene
5180	1,2,3-Trichloropropane
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)



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4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
4615	1,3-Dichlorobenzene
4660	1,3-Dichloropropane
4620	1,4-Dichlorobenzene
4915	1,4-Isopropyltoluene
4665	2,2-Dichloropropane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4500	2-Chloroethyl vinyl ether
4535	2-Chlorotoluene
4860	2-Hexanone
4540	4-Chlorotoluene
4995	4-Methyl-2-pentanone (MIBK)
4315	Acetone
4325	Acrolein (Propenal)
4340	Acrylonitrile
4375	Benzene
4385	Bromobenzene
4390	Bromochloromethane
4395	Bromodichloromethane
255	Bromoethane (Ethyl Bromide)
4400	Bromoform
4950	Bromomethane (Methyl bromide)
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4485	Chloroethane
4505	Chloroform
4960	Chloromethane (Methyl chloride)
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4560	Cyclohexanone
4575	Dibromochloromethane
4595	Dibromomethane
4625	Dichlorodifluoromethane
4650	Dichloromethane (DCM, Methylene chloride)
4765	Ethylbenzene
4835	Hexachlorobutadiene
4870	Iodomethane (Methyl iodide)
4900	Isopropylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
5255	p-Xylene
4440	sec-Butylbenzene



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- 6700 alpha-Terpineol
- 5545 Aniline
- 5555 Anthracene
- 123 Azobenzene
- 5575 Benzo[a]anthracene
- 5580 Benzo[a]pyrene
- 5585 Benzo[b]fluoranthene
- 5590 Benzo[g,h,i]perylene
- 5600 Benzo[k]fluoranthene
- 5610 Benzoic acid
- 5630 Benzyl alcohol
- 5760 bis(2-Chloroethoxy)methane
- 5765 bis(2-Chloroethyl)ether
- 6255 bis(2-Ethylhexyl)phthalate (DEHP)
- 5670 Butyl benzyl phthalate
- 257 Butylated Hydroxy Toluene (BHT)
- 5680 Carbazole
- 5855 Chrysene
- 5895 Dibenz[a,h]anthracene
- 5905 Dibenzofuran
- 260 Dibutylphenyl Phosphahate
- 6070 Diethyl phthalate
- 6135 Dimethyl phthalate
- 5925 Di-n-butyl phthalate
- 6200 Di-n-octyl phthalate
- 3800 Ethyl parathion
- 6265 Fluoranthene
- 6270 Fluorene
- 6275 Hexachlorobenzene
- 4835 Hexachlorobutadiene
- 6285 Hexachlorocyclopentadiene
- 4840 Hexachloroethane
- 6315 Indeno[1,2,3-cd]pyrene
- 6320 Isophorone
- 7825 Methyl parathion (Parathion, methyl)
- 5005 Naphthalene
- 5015 Nitrobenzene
- 6530 n-Nitrosodimethylamine
- 6545 n-Nitrosodi-n-propylamine
- 6535 n-Nitrosodiphenylamine
- 6605 Pentachlorophenol
- 6615 Phenanthrene
- 6625 Phenol
- 6665 Pyrene
- 5095 Pyridine
- 6670 Quinoline
- 261 Tributyl Phosphate
- 258 Triphenyl Phosphate

EPA 9010C

686

Total and Amenable Cyanide by Distillation and UV-Vis

Analyte Code

Analyte

1510

Amenable cyanide

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1645	Total cyanide		
EPA 9014		10193803	Titrimetric and Manual Spectrophotometric Determinative Methods for Cy
<u>Analyte Code</u>	<u>Analyte</u>		
1635	Cyanide		
EPA 9036		10196404	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
<u>Analyte Code</u>	<u>Analyte</u>		
2000	Sulfate		
EPA 9040C		688	pH Electrometric Measurement
<u>Analyte Code</u>	<u>Analyte</u>		
1900	pH		
EPA 9050A		10198808	Specific Conductance
<u>Analyte Code</u>	<u>Analyte</u>		
1610	Conductivity		
EPA 9056A		10199607	Determination of Inorganic Anions by Ion Chromatography
<u>Analyte Code</u>	<u>Analyte</u>		
1540	Bromide		
1575	Chloride		
1730	Fluoride		
1810	Nitrate as N		
1840	Nitrite as N		
1870	Orthophosphate as P		
2000	Sulfate		
EPA 9060A		690	Total Organic Carbon
<u>Analyte Code</u>	<u>Analyte</u>		
2040	Total Organic Carbon		
EPA 9065		10200405	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)
<u>Analyte Code</u>	<u>Analyte</u>		
1905	Total Phenolics		
EPA 9214		10206403	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Se
<u>Analyte Code</u>	<u>Analyte</u>		
1730	Fluoride		
EPA 9251		10207406	Chloride (Colorimetric, Automated Ferricyanide AAII)
<u>Analyte Code</u>	<u>Analyte</u>		
1575	Chloride		
NWTPH-DX		104	Oregon DEQ TPH Diesel Range
<u>Analyte Code</u>	<u>Analyte</u>		
9369	Diesel range organics (DRO)		
NWTPH-GX		105	Oregon DEQ TPH Gasoline Range Organics by GC/PID Purge & Trap
<u>Analyte Code</u>	<u>Analyte</u>		
9408	Gasoline range organics (GRO)		
NWTPH-HCID		107	Oregon DEQ Total Petroleum Hydrocarbon ID
<u>Analyte Code</u>	<u>Analyte</u>		
2050	Total Petroleum Hydrocarbons (TPH)		
SM 19th ED 2120 B		370	Color by Visual Comparison
<u>Analyte Code</u>	<u>Analyte</u>		
1605	Color		
SM 19th ED 2130 B		371	Turbidity by Nephelometric Determination
<u>Analyte Code</u>	<u>Analyte</u>		
2055	Turbidity		
SM 19th ED 2310 B		539	Acidity by Titration
<u>Analyte Code</u>	<u>Analyte</u>		

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1500	Acidity, as CaCO3		
SM 19th ED 2340 B		374	Hardness by calculation
<u>Analyte Code</u>	<u>Analyte</u>		
1750	Hardness		
SM 19th ED 2510 B		376	Conductivity by Probe
<u>Analyte Code</u>	<u>Analyte</u>		
1610	Conductivity		
SM 19th ED 2540 B		621	Total Solids
<u>Analyte Code</u>	<u>Analyte</u>		
1950	Residue-total		
SM 19th ED 2540 C		377	Total Dissolved Solids
<u>Analyte Code</u>	<u>Analyte</u>		
1955	Residue-filterable (TDS)		
SM 19th ED 2540 D		623	Total Suspended Solids
<u>Analyte Code</u>	<u>Analyte</u>		
1960	Residue-nonfilterable (TSS)		
SM 19th ED 2540 F		625	Settleable Solids
<u>Analyte Code</u>	<u>Analyte</u>		
1965	Residue-settleable		
SM 19th ED 3120 B		385	Metals by Inductively Coupled Plasma
<u>Analyte Code</u>	<u>Analyte</u>		
1000	Aluminum		
1005	Antimony		
1010	Arsenic		
1015	Barium		
1020	Beryllium		
1025	Boron		
1030	Cadmium		
1035	Calcium		
1040	Chromium		
1050	Cobalt		
1055	Copper		
1070	Iron		
1075	Lead		
1085	Magnesium		
1090	Manganese		
1100	Molybdenum		
1105	Nickel		
1125	Potassium		
1140	Selenium		
1145	Silicon		
1150	Silver		
1155	Sodium		
1160	Strontium		
1165	Thallium		
1175	Tin		
1180	Titanium		
1185	Vanadium		
1190	Zinc		
SM 19th ED 3500-Cr D		578	Chromium by Colorimetric Method
<u>Analyte Code</u>	<u>Analyte</u>		

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1040	Chromium		
SM 19th ED 4110 B		388	Anions by Ion Chromatography with Chemical Suppression of Eluent
<u>Analyte Code</u>	<u>Analyte</u>		
1540	Bromide		
1575	Chloride		
1730	Fluoride		
1810	Nitrate as N		
1840	Nitrite as N		
1870	Orthophosphate as P		
2000	Sulfate		
SM 19th ED 4500-CI E		404	Chloride by Automated Ferricyanide Method
<u>Analyte Code</u>	<u>Analyte</u>		
1575	Chloride		
SM 19th ED 4500-CN G		392	Cyanide Amenable to Chlorination after Distillation
<u>Analyte Code</u>	<u>Analyte</u>		
1510	Amenable cyanide		
SM 19th ED 4500-F C		394	Fluoride by Ion Selective Electrode
<u>Analyte Code</u>	<u>Analyte</u>		
1730	Fluoride		
SM 19th ED 4500-H+ B		412	pH by Probe
<u>Analyte Code</u>	<u>Analyte</u>		
1900	pH		
SM 19th ED 4500-NH3 D		413	Ammonia by Selective Ion Probe
<u>Analyte Code</u>	<u>Analyte</u>		
1515	Ammonia as N		
SM 19th ED 4500-NH3 G		414	Ammonia by Automated Phenate
<u>Analyte Code</u>	<u>Analyte</u>		
1515	Ammonia as N		
SM 19th ED 4500-NO3 F		419	Nitrate Nitrogen by Automated Cadmium Reduction Method
<u>Analyte Code</u>	<u>Analyte</u>		
1810	Nitrate as N		
SM 19th ED 4500-O C		613	Dissolved Oxygen by Azide Modification
<u>Analyte Code</u>	<u>Analyte</u>		
1880	Oxygen, dissolved		
SM 19th ED 4500-P B 5		617	Phosphorus by Persulfate Digestion Method
<u>Analyte Code</u>	<u>Analyte</u>		
1910	Phosphorus, total		
SM 19th ED 4500-S2 D		629	Sulfide by Methylene Blue Method
<u>Analyte Code</u>	<u>Analyte</u>		
2005	Sulfide		
SM 19th ED 4500-SO4 F		402	Sulfate by Automated Methylthymol Blue
<u>Analyte Code</u>	<u>Analyte</u>		
2000	Sulfate		
SM 19th ED 5210 B		563	Biochemical Oxygen Demand, 5-Day
<u>Analyte Code</u>	<u>Analyte</u>		
1530	Biochemical oxygen demand		
SM 19th ED 5220 D		574	Chemical Oxygen Demand by Closed Reflux and Colorimetric Determinat
<u>Analyte Code</u>	<u>Analyte</u>		
1565	Chemical oxygen demand		
SM 19th ED 5310 B		424	Total Organic Carbon by Combustion Infra-red Method
<u>Analyte Code</u>	<u>Analyte</u>		

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2040	Total Organic Carbon	641	Volatile Organic Compounds by purge and trap GC/MS
SM 19th ED 6210 B			
Analyte Code	Analyte		
5105	1,1,1,2-Tetrachloroethane		
5160	1,1,1-Trichloroethane		
5110	1,1,2,2-Tetrachloroethane		
5195	1,1,2-Trichloro-1,2,2-trifluoroethane		
5165	1,1,2-Trichloroethane		
4630	1,1-Dichloroethane		
4640	1,1-Dichloroethylene		
4670	1,1-Dichloropropene		
5150	1,2,3-Trichlorobenzene		
5180	1,2,3-Trichloropropane		
5155	1,2,4-Trichlorobenzene		
5210	1,2,4-Trimethylbenzene		
4570	1,2-Dibromo-3-chloropropane (DBCP)		
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)		
4610	1,2-Dichlorobenzene		
4635	1,2-Dichloroethane		
4655	1,2-Dichloropropane		
5215	1,3,5-Trimethylbenzene		
4615	1,3-Dichlorobenzene		
4660	1,3-Dichloropropane		
4620	1,4-Dichlorobenzene		
4665	2,2-Dichloropropane		
4410	2-Butanone (Methyl ethyl ketone, MEK)		
4500	2-Chloroethyl vinyl ether		
4535	2-Chlorotoluene		
4860	2-Hexanone		
4540	4-Chlorotoluene		
5870	4-Isopropyltoluene		
4995	4-Methyl-2-pentanone (MIBK)		
4315	Acetone		
4325	Acrolein (Propenal)		
4340	Acrylonitrile		
4375	Benzene		
4385	Bromobenzene		
4390	Bromochloromethane		
4395	Bromodichloromethane		
255	Bromoethane (Ethyl Bromide)		
4400	Bromoform		
4950	Bromomethane (Methyl bromide)		
4450	Carbon disulfide		
4455	Carbon tetrachloride		
4475	Chlorobenzene		
4485	Chloroethane		
4505	Chloroform		
105	Chloromethane		
4645	cis-1,2-Dichloroethylene		
4680	cis-1,3-Dichloropropene		
4560	Cyclohexanone		

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4575	Dibromochloromethane
4595	Dibromomethane
4625	Dichlorodifluoromethane
4650	Dichloromethane (DCM, Methylene chloride)
4765	Ethylbenzene
4835	Hexachlorobutadiene
4870	Iodomethane (Methyl iodide)
4900	Isopropylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
4605	trans-1,4-Dichloro-2-butene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane
5225	Vinyl acetate
5235	Vinyl chloride

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<b>SM 19th ED 6220 B</b>	<b>642</b>	<b>Volatile Aromatic Organic Compounds by GC/PID Purge &amp; Trap</b>
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<u>Analyte Code</u>	<u>Analyte</u>
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5250	o-Xylene

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<b>SM 19th ED 6630 B</b>	<b>646</b>	<b>Organochlorine Pesticides by Liquid/Liquid Extraction and GC/ECD</b>
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<u>Analyte Code</u>	<u>Analyte</u>
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
7685	Heptachlor



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7690	Heptachlor epoxide
7810	Methoxychlor
7870	Mirex
8250	Toxaphene (Chlorinated camphene)

**SM 19th ED 6630 C 648 Organochlorine Pesticides and PCBs by Liquid/Liquid Extraction and GC/**

<u>Analyte Code</u>	<u>Analyte</u>
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
184	Aroclor-1262 (PCB 1262)
103	Aroclor-1268 (PCB-1268)
7300	Chlorpyrifos
7925	cis-Nonachlor
8550	Dacthal (DCPA)
7735	Kelthane (Diclofol)
7825	Methyl parathion (Parathion, methyl)
7870	Mirex
274	Oxadiazon
3890	Oxychlorthane
7950	Parathion
7955	Parathion, ethyl
8250	Toxaphene (Chlorinated camphene)
7910	trans Nanochlor
8295	Trifluralin (Treflan)

**WA EPH 154 Extractable Petroleum Hydrocarbons**

<u>Analyte Code</u>	<u>Analyte</u>
222	EPH Aliphatic >C10-C12
223	EPH Aliphatic >C12-C16
224	EPH Aliphatic >C16-C21
225	EPH Aliphatic >C21-C34
221	EPH Aliphatic C8-C10
217	EPH Aromatic >C10-C12
218	EPH Aromatic >C12-C16
219	EPH Aromatic >C16-C21
220	EPH Aromatic >C21-C34
216	EPH Aromatic C8-C10

**WA VPH 155 Volatile Petroleum Hydrocarbons (VPH) by GC/PID Purge & Trap**

<u>Analyte Code</u>	<u>Analyte</u>
229	VPH Aliphatic >C10-C12
227	VPH Aliphatic >C6-C8
228	VPH Aliphatic >C8-C10
226	VPH Aliphatic C5-C6
231	VPH Aromatic >C10-C12
232	VPH Aromatic >C12-C13

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VPH/Aromatic >C8-C10

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<b>MATRIX: Solids</b>		
<b>Reference</b>	<b>Code</b>	<b>Description</b>
EPA 1311	10118806	Toxicity Characteristic Leaching Procedure
<u>Analyte Code</u> <u>Analyte</u>		
125		Extraction/Preparation
EPA 1312	10119003	Synthetic Precipitation Leaching Procedure
<u>Analyte Code</u> <u>Analyte</u>		
125		Extraction/Preparation
EPA 150.1	10008409	pH - Electrometric Measurement
<u>Analyte Code</u> <u>Analyte</u>		
1900		pH
EPA 160.3	10010001	Total Solids, dried @ 103-105 C.
<u>Analyte Code</u> <u>Analyte</u>		
1950		Residue-total
EPA 160.4	10010409	Total Volatile Solids, ignition @ 550 C.
<u>Analyte Code</u> <u>Analyte</u>		
1970		Residue-volatile
EPA 1664A	10127807	N-Hexane Extractable Material
<u>Analyte Code</u> <u>Analyte</u>		
109		Hexane Extractable Material
EPA 200.9	10015608	Metals by Graphite Atomic Absorption
<u>Analyte Code</u> <u>Analyte</u>		
1005		Antimony
1010		Arsenic
1030		Cadmium
1075		Lead
1140		Selenium
1150		Silver
1165		Thallium
EPA 245.5	10037602	Mercury in Sediment by Cold Vapor Atomic Absorption
<u>Analyte Code</u> <u>Analyte</u>		
1095		Mercury
EPA 300.0	10053200	Inorganic Anions in water by Ion Chromatography
<u>Analyte Code</u> <u>Analyte</u>		
1540		Bromide
1575		Chloride
1730		Fluoride
1810		Nitrate as N
1840		Nitrite as N
1870		Orthophosphate as P
2000		Sulfate
EPA 3050B	10135601	Acid Digestion of Sediments, Sludges, and soils
<u>Analyte Code</u> <u>Analyte</u>		
125		Extraction/Preparation
EPA 325.2	10057202	Chloride - Colorimetric, automated Ferricyanide,II
<u>Analyte Code</u> <u>Analyte</u>		
1575		Chloride
EPA 335.1	10060001	Cyanides, amenable to chlorination
<u>Analyte Code</u> <u>Analyte</u>		
1510		Amenable cyanide

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EPA 335.2	10060409	Cyanide - Titrimetric; Spectrophotometric
<u>Analyte Code</u>	<u>Analyte</u>	
1635	Cyanide	
EPA 340.2	10062201	Fluoride - Ion selective electrode
<u>Analyte Code</u>	<u>Analyte</u>	
1730	Fluoride	
EPA 350.1	10063602	Ammonia Nitrogen - Colorimetric, Auto Phenate
<u>Analyte Code</u>	<u>Analyte</u>	
1515	Ammonia as N	
EPA 351.4	10066203	Kjeldahl Nitrogen - Ion Selective Electrode
<u>Analyte Code</u>	<u>Analyte</u>	
1795	Kjeldahl nitrogen - total	
EPA 353.2	10067604	Nitrate/Nitrite Nitrogen - Automated, Cadmium
<u>Analyte Code</u>	<u>Analyte</u>	
1820	Nitrate+nitrite	
EPA 3540C	10140202	Soxhlet Extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3545A	10141001	Pressurized Fluid Extraction (PFE)
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3550C	10142004	Ultrasonic Extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3630C	10146802	Silica gel cleanup
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3640A	10147203	Gel Preparation Cleanup
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 365.2	10070403	Phosphorous - Colorimetric, single reagent
<u>Analyte Code</u>	<u>Analyte</u>	
1870	Orthophosphate as P	
EPA 3660B	10148400	Sulfur cleanup
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 376.1	10074201	Sulfide - Titrimetric, Iodine.
<u>Analyte Code</u>	<u>Analyte</u>	
2005	Sulfide	
EPA 376.2	10074609	Sulfide - Colorimetric, Methylene Blue.
<u>Analyte Code</u>	<u>Analyte</u>	
2005	Sulfide	
EPA 5000	10152600	Sample Preparation for Volatile Organics
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 5035	10154004	Closed-System Purge-and-Trap and Extraction for Volatile Organics in So
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 6010B	10155609	ICP - AES
<u>Analyte Code</u>	<u>Analyte</u>	
1000	Aluminum	

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1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1035	Calcium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1085	Magnesium
1090	Manganese
1100	Molybdenum
1105	Nickel
1125	Potassium
1140	Selenium
1145	Silicon
1150	Silver
1155	Sodium
1160	Strontium
1165	Thallium
1175	Tin
1180	Titanium
1185	Vanadium
1190	Zinc

EPA 7041	10158404	Antimony by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1005	Antimony	
EPA 7060A	10158802	Arsenic by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1010	Arsenic	
EPA 7131A	10161203	Cadmium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1030	Cadmium	
EPA 7196A	10162400	Chromium Hexavalent colorimetric
<u>Analyte Code</u>	<u>Analyte</u>	
1045	Chromium VI	
EPA 7421	10164600	Lead by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1075	Lead	
EPA 7471B	10166402	Mercury by Cold Vapor Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1095	Mercury	
EPA 7740	10168602	Selenium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1140	Selenium	
EPA 7761	10169809	Silver by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1150	Silver	

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EPA 7841	10170602	Thallium by Graphite Furnace Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1165	Thallium	
EPA 8000B	10172200	Determinative Chromatographic Separations
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 8021B	10174808	Aromatic and Halogenated Volatiles by GC with PID and/or ECD Purge &
<u>Analyte Code</u>	<u>Analyte</u>	
4375	Benzene	
4765	Ethylbenzene	
5240	m+p-xylene	
5000	Methyl tert-butyl ether (MTBE)	
5250	o-Xylene	
5140	Toluene	
EPA 8081B	10178800	Organochlorine Pesticides by GC/ECD
<u>Analyte Code</u>	<u>Analyte</u>	
8580	2,4'-DDD	
8585	2,4'-DDE	
8590	2,4'-DDT	
7355	4,4'-DDD	
7360	4,4'-DDE	
7365	4,4'-DDT	
7025	Aldrin	
7110	alpha-BHC (alpha-Hexachlorocyclohexane)	
7240	alpha-Chlordane	
7115	beta-BHC (beta-Hexachlorocyclohexane)	
7250	Chlordane (tech.)	
7300	Chlorpyrifos	
7925	cis-Nonachlor	
8550	Dacthal (DCPA)	
7105	delta-BHC	
7470	Dieldrin	
7510	Endosulfan I	
7515	Endosulfan II	
7520	Endosulfan sulfate	
7540	Endrin	
7530	Endrin aldehyde	
7535	Endrin-ketone	
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	
7685	Heptachlor	
7690	Heptachlor epoxide	
6275	Hexachlorobenzene	
7735	Kelthane (Diclofol)	
7810	Methoxychlor	
7825	Methyl parathion (Parathion, methyl)	
7870	Mirex	
274	Oxadiazon	
3890	Oxychlordane	
7950	Parathion	
7955	Parathion, ethyl	
7910	trans Nanochlor	

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8295		Trifluralin (Treflan)	
EPA 8082	10179007	Polychlorinated Biphenyls (PCBs) by GC/ECD	
<u>Analyte Code</u>	<u>Analyte</u>		
8880	Aroclor-1016 (PCB-1016)		
8885	Aroclor-1221 (PCB-1221)		
8890	Aroclor-1232 (PCB-1232)		
8895	Aroclor-1242 (PCB-1242)		
8900	Aroclor-1248 (PCB-1248)		
8905	Aroclor-1254 (PCB-1254)		
8910	Aroclor-1260 (PCB-1260)		
184	Aroclor-1262 (PCB 1262)		
103	Aroclor-1268 (PCB-1268)		
EPA 8151A	10183207	Chlorinated Herbicides by GC/ECD	
<u>Analyte Code</u>	<u>Analyte</u>		
8655	2,4,5-T		
8545	2,4-D		
8560	2,4-DB		
6500	4-Nitrophenol		
8555	Dalapon		
8595	Dicamba		
8605	Dichloroprop (Dichlorprop)		
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)		
7775	MCPA		
7780	MCPP		
6605	Pentachlorophenol		
8650	Silvex (2,4,5-TP)		
EPA 8260B	10184802	Volatile Organic Compounds by purge and trap GC/MS	
<u>Analyte Code</u>	<u>Analyte</u>		
5105	1,1,1,2-Tetrachloroethane		
5160	1,1,1-Trichloroethane		
5110	1,1,1,2-Tetrachloroethane		
5195	1,1,2-Trichloro-1,2,2-trifluoroethane		
5165	1,1,2-Trichloroethane		
4630	1,1-Dichloroethane		
4640	1,1-Dichloroethylene		
4670	1,1-Dichloropropene		
5150	1,2,3-Trichlorobenzene		
5180	1,2,3-Trichloropropane		
5155	1,2,4-Trichlorobenzene		
5210	1,2,4-Trimethylbenzene		
4570	1,2-Dibromo-3-chloropropane (DBCP)		
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)		
4610	1,2-Dichlorobenzene		
4635	1,2-Dichloroethane		
4655	1,2-Dichloropropane		
5215	1,3,5-Trimethylbenzene		
4615	1,3-Dichlorobenzene		
4660	1,3-Dichloropropane		
4620	1,4-Dichlorobenzene		
4665	2,2-Dichloropropane		
4410	2-Butanone (Methyl ethyl ketone, MEK)		

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- 4500 2-Chloroethyl vinyl ether
- 4535 2-Chlorotoluene
- 4860 2-Hexanone
- 4540 4-Chlorotoluene
- 4995 4-Methyl-2-pentanone (MIBK)
- 4315 Acetone
- 4325 Acrolein (Propenal)
- 4340 Acrylonitrile
- 4375 Benzene
- 4385 Bromobenzene
- 4390 Bromochloromethane
- 4395 Bromodichloromethane
- 255 Bromoethane (Ethyl Bromide)
- 4400 Bromoform
- 4950 Bromomethane (Methyl bromide)
- 4450 Carbon disulfide
- 4455 Carbon tetrachloride
- 4475 Chlorobenzene
- 4485 Chloroethane
- 4505 Chloroform
- 4960 Chloromethane (Methyl chloride)
- 4645 cis-1,2-Dichloroethylene
- 4680 cis-1,3-Dichloropropene
- 4560 Cyclohexanone
- 4575 Dibromochloromethane
- 4595 Dibromomethane
- 4625 Dichlorodifluoromethane
- 4765 Ethylbenzene
- 4835 Hexachlorobutadiene
- 4870 Iodomethane (Methyl iodide)
- 4900 Isopropylbenzene
- 5240 m+p-xylene
- 5000 Methyl tert-butyl ether (MTBE)
- 4975 Methylene chloride
- 5005 Naphthalene
- 4435 n-Butylbenzene
- 5090 n-Propylbenzene
- 5250 o-Xylene
- 4910 p-Isopropyltoluene
- 4440 sec-Butylbenzene
- 5100 Styrene
- 4445 tert-Butylbenzene
- 5115 Tetrachloroethylene (Perchloroethylene)
- 5140 Toluene
- 4700 trans-1,2-Dichloroethylene
- 4685 trans-1,3-Dichloropropylene
- 4605 trans-1,4-Dichloro-2-butene
- 5170 Trichloroethene (Trichloroethylene)
- 5175 Trichlorofluoromethane
- 5225 Vinyl acetate
- 5235 Vinyl chloride



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EPA 8270D	10186002	SemiVolatile Organic compounds by GC/MS
Analyte Code	Analyte	
6715	1,2,4,5-Tetrachlorobenzene	
5155	1,2,4-Trichlorobenzene	
4610	1,2-Dichlorobenzene	
4615	1,3-Dichlorobenzene	
4620	1,4-Dichlorobenzene	
6380	1-Methylnaphthalene	
183	2,2'-Oxybis(1-chloropropane)	
6730	2,3,4,5-Tetrachlorophenol	
6735	2,3,4,6-Tetrachlorophenol	
6740	2,3,5,6-Tetrachlorophenol	
6835	2,4,5-Trichlorophenol	
6840	2,4,6-Trichlorophenol	
6000	2,4-Dichlorophenol	
6130	2,4-Dimethylphenol	
6175	2,4-Dinitrophenol	
6185	2,4-Dinitrotoluene (2,4-DNT)	
6190	2,6-Dinitrotoluene (2,6-DNT)	
5795	2-Chloronaphthalene	
5800	2-Chlorophenol	
6385	2-Methylnaphthalene	
6400	2-Methylphenol (o-Cresol)	
6460	2-Nitroaniline	
6490	2-Nitrophenol	
5945	3,3'-Dichlorobenzidine	
6815	3,4,5-Trichloroguaiacol	
268	3-beta-Coprostanol	
146	3-Chlorophenol	
6465	3-Nitroaniline	
7365	4,4'-DDT	
6825	4,5,6-Trichloroguaiacol	
264	4,5-Dichloro-2-methoxyphenol	
6140	4,6-Dinitro-2-methylphenol	
5660	4-Bromophenyl phenyl ether	
5700	4-Chloro-3-methylphenol	
5745	4-Chloroaniline	
5825	4-Chlorophenyl phenylether	
5870	4-Isopropyltoluene	
6410	4-Methylphenol (p-Cresol)	
6470	4-Nitroaniline	
6500	4-Nitrophenol	
271	4-Nonylphenol	
6115	7,12-Dimethylbenz[a]anthracene	
5500	Acenaphthene	
5505	Acenaphthylene	
5510	Acetophenone	
6700	alpha-Terpineol	
5545	Aniline	
5555	Anthracene	
123	Azobenzene	

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6745	n-Tetradecane		
272	Pentabromodiphenyl Ether		
6605	Pentachlorophenol		
267	Perylene		
6615	Phenanthrene		
6625	Phenol		
6665	Pyrene		
5095	Pyridine		
6670	Quinoline		
266	Retene		
6725	Tetrachloroguaiacol		
273	Tetraethyl Tin		
261	Tributyl Phosphate		
258	Triphenyl Phosphate		
<b>EPA 9010C</b>		<b>686</b>	<b>Total and Amenable Cyanide by Distillation and UV-Vis</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1510	Amenable cyanide		
1645	Total cyanide		
<b>EPA 9014</b>		<b>10193803</b>	<b>Titrimetric and Manual Spectrophotometric Determinative Methods for Cy</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1635	Cyanide		
<b>EPA 9030B</b>		<b>10195605</b>	<b>Acid-Soluble and Acid-Insoluble sulfides: Distillation</b>
<u>Analyte Code</u>	<u>Analyte</u>		
2005	Sulfide		
<b>EPA 9045D</b>		<b>689</b>	<b>Soil and Waste pH</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1900	pH		
<b>EPA 9056A</b>		<b>10199607</b>	<b>Determination of Inorganic Anions by Ion Chromatography</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1540	Bromide		
1575	Chloride		
1730	Fluoride		
1810	Nitrate as N		
1840	Nitrite as N		
1870	Orthophosphate as P		
2000	Sulfate		
<b>EPA 9065</b>		<b>10200405</b>	<b>Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1905	Total Phenolics		
<b>EPA 9071B</b>		<b>10201602</b>	<b>Oil and Grease Extraction Method for sludge and sediment samples</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1860	Oil & Grease		
<b>EPA 9080</b>		<b>10203200</b>	<b>Cation-Exchange Capacity of Soils (Ammonium Acetate)</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1560	Cation exchange capacity		
<b>EPA 9081</b>		<b>10203404</b>	<b>Cation-Exchange Capacity of Soils (Sodium Acetate)</b>
<u>Analyte Code</u>	<u>Analyte</u>		
1560	Cation exchange capacity		
<b>EPA 9095B</b>		<b>692</b>	<b>Paint Filter Liquids Test</b>
<u>Analyte Code</u>	<u>Analyte</u>		
125	Extraction/Preparation		

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5595	Benzidine
5575	Benzo[a]anthracene
5580	Benzo[a]pyrene
5585	Benzo[b]fluoranthene
5605	Benzo[e]pyrene
5590	Benzo[g,h,i]perylene
5600	Benzo[k]fluoranthene
5610	Benzoic acid
5630	Benzyl alcohol
262	beta-Pinene
270	beta-Sitosterol
5640	Biphenyl
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl)ether
6255	bis(2-Ethylhexyl)phthalate (DEHP)
5670	Butyl benzyl phthalate
257	Butylated Hydroxy Toluene (BHT)
259	Butyldiphenyl Phosphate
5675	Caffeine
5680	Carbazole
7300	Chlorpyrifos
269	Cholesterol
5855	Chrysene
5895	Dibenz[a,h]anthracene
5905	Dibenzofuran
5910	Dibenzothiophene
260	Dibutylphenyl Phosphahate
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
263	Diphenyl Oxide
7565	Ethion
3800	Ethyl parathion
6265	Fluoranthene
6270	Fluorene
265	Guaiacol
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
6285	Hexachlorocyclopentadiene
4840	Hexachloroethane
6315	Indeno[1,2,3-cd]pyrene
6320	Isophorone
7735	Kelthane (Diclofol)
7825	Methyl parathion (Parathion, methyl)
5005	Naphthalene
6300	n-Hexadecane
5015	Nitrobenzene
6530	n-Nitrosodimethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine

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4765	Ethylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5250	o-Xylene
5140	Toluene

WA EPH	154	Extractable Petroleum Hydrocarbons
<u>Analyte Code</u>	<u>Analyte</u>	
222	EPH Aliphatic >C10-C12	
223	EPH Aliphatic >C12-C16	
224	EPH Aliphatic >C16-C21	
225	EPH Aliphatic >C21-C34	
221	EPH Aliphatic C8-C10	
217	EPH Aromatic >C10-C12	
218	EPH Aromatic >C12-C16	
219	EPH Aromatic >C16-C21	
220	EPH Aromatic >C21-C34	
216	EPH Aromatic C8-C10	

WA VPH	155	Volatile Petroleum Hydrocarbons (VPH) by GC/PID Purge & Trap
<u>Analyte Code</u>	<u>Analyte</u>	
229	VPH Aliphatic >C10-C12	
227	VPH Aliphatic >C6-C8	
228	VPH Aliphatic >C8-C10	
226	VPH Aliphatic C5-C6	
231	VPH Aromatic >C10-C12	
232	VPH Aromatic >C12-C13	
230	VPH Aromatic >C8-C10	

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EPA 9214	10206403	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Sele
<u>Analyte Code</u> 1730	<u>Analyte</u> Fluoride	
EPA 9251	10207406	Chloride (Colorimetric, Automated Ferricyanide AAll)
<u>Analyte Code</u> 1575	<u>Analyte</u> Chloride	
NWTPH-DX	104	Oregon DEQ TPH Diesel Range
<u>Analyte Code</u> 9369	<u>Analyte</u> Diesel range organics (DRO)	
NWTPH-GX	105	Oregon DEQ TPH Gasoline Range Organics by GC/PID Purge & Trap
<u>Analyte Code</u> 9408	<u>Analyte</u> Gasoline range organics (GRO)	
NWTPH-HCID	107	Oregon DEQ Total Petroleum Hydrocarbon ID
<u>Analyte Code</u> 2050	<u>Analyte</u> Total Petroleum Hydrocarbons (TPH)	
SM 19th ED 2540 B	621	Total Solids
<u>Analyte Code</u> 1950	<u>Analyte</u> Residue-total	
SM 19th ED 4110 B	388	Anions by Ion Chromatography with Chemical Suppression of Eluent
<u>Analyte Code</u> 1540	<u>Analyte</u> Bromide	
1575	Chloride	
1730	Fluoride	
1810	Nitrate as N	
1840	Nitrite as N	
1870	Orthophosphate as P	
2000	Sulfate	
SM 19th ED 4500-CI E	404	Chloride by Automated Ferricyanide Method
<u>Analyte Code</u> 1575	<u>Analyte</u> Chloride	
SM 19th ED 4500-CN G	392	Cyanide Amenable to Chlorination after Distillation
<u>Analyte Code</u> 1510	<u>Analyte</u> Amenable cyanide	
SM 19th ED 4500-F C	394	Fluoride by Ion Selective Electrode
<u>Analyte Code</u> 1730	<u>Analyte</u> Fluoride	
SM 19th ED 4500-H+ B	412	pH by Probe
<u>Analyte Code</u> 1900	<u>Analyte</u> pH	
SM 19th ED 4500-NH3 D	413	Ammonia by Selective Ion Probe
<u>Analyte Code</u> 1515	<u>Analyte</u> Ammonia as N	
SM 19th ED 4500-S2 D	629	Sulfide by Methylene Blue Method
<u>Analyte Code</u> 2005	<u>Analyte</u> Sulfide	
SM 19th ED 5310 B	424	Total Organic Carbon by Combustion Infra-red Method
<u>Analyte Code</u> 2040	<u>Analyte</u> Total Organic Carbon	
SM 19th ED 6220 B	642	Volatile Aromatic Organic Compounds by GC/PID Purge & Trap
<u>Analyte Code</u> 4375	<u>Analyte</u> Benzene	

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Cusotmers: Please verify the current accreditation standing with ORELAP.

<b>MATRIX: Biological Tissue</b>		
<b>Reference</b>	<b>Code</b>	<b>Description</b>
EPA 3540C	10140202	Soxhlet Extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3545A	10141001	Pressurized Fluid Extraction (PFE)
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3550C	10142004	Ultrasonic Extraction
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 3640A	10147203	Gel Preparation Cleanup
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 5000	10152600	Sample Preparation for Volatile Organics
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 5035	10154004	Closed-System Purge-and-Trap and Extraction for Volatile Organics in So
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 6010B	10155609	ICP - AES
<u>Analyte Code</u>	<u>Analyte</u>	
1000	Aluminum	
1005	Antimony	
1010	Arsenic	
1015	Barium	
1020	Beryllium	
1025	Boron	
1030	Cadmium	
1035	Calcium	
1040	Chromium	
1050	Cobalt	
1055	Copper	
1070	Iron	
1075	Lead	
1085	Magnesium	
1090	Manganese	
1100	Molybdenum	
1105	Nickel	
1125	Potassium	
1140	Selenium	
1145	Silicon	
1150	Silver	
1155	Sodium	
1160	Strontium	
1165	Thallium	
1175	Tin	
1180	Titanium	
1185	Vanadium	
1190	Zinc	

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 Cusotmers: Please verify the current accreditation standing with ORELAP.

EPA 7471B	10166402	Mercury by Cold Vapor Atomic Absorption
<u>Analyte Code</u>	<u>Analyte</u>	
1095	Mercury	
EPA 8000B	10172200	Determinative Chromatographic Separations
<u>Analyte Code</u>	<u>Analyte</u>	
125	Extraction/Preparation	
EPA 8081B	10178800	Organochlorine Pesticides by GC/ECD
<u>Analyte Code</u>	<u>Analyte</u>	
8580	2,4'-DDD	
8585	2,4'-DDE	
8590	2,4'-DDT	
7355	4,4'-DDD	
7360	4,4'-DDE	
7365	4,4'-DDT	
7025	Aldrin	
7110	alpha-BHC (alpha-Hexachlorocyclohexane)	
7240	alpha-Chlordane	
7115	beta-BHC (beta-Hexachlorocyclohexane)	
7250	Chlordane (tech.)	
7300	Chlorpyrifos	
7925	cis-Nonachlor	
8550	Dacthal (DCPA)	
7105	delta-BHC	
7470	Dieldrin	
7510	Endosulfan I	
7515	Endosulfan II	
7520	Endosulfan sulfate	
7540	Endrin	
7530	Endrin aldehyde	
7535	Endrin ketone	
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	
7685	Heptachlor	
7690	Heptachlor epoxide	
6275	Hexachlorobenzene	
4835	Hexachlorobutadiene	
7735	Kelthane (Diclofol)	
7810	Methoxychlor	
7825	Methyl parathion (Parathion, methyl)	
7870	Mirex	
274	Oxadiazon	
3890	Oxychlordane	
7950	Parathion	
7955	Parathion, ethyl	
8250	Toxaphene (Chlorinated camphene)	
7910	trans Nanochlor	
8295	Trifluralin (Treflan)	
EPA 8082	10179007	Polychlorinated Biphenyls (PCBs) by GC/ECD
<u>Analyte Code</u>	<u>Analyte</u>	
8880	Aroclor-1016 (PCB-1016)	
8885	Aroclor-1221 (PCB-1221)	
8890	Aroclor-1232 (PCB-1232)	

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Cusotmers: Please verify the current accreditation standing with ORELAP.

- 8895 Aroclor-1242 (PCB-1242)
- 8900 Aroclor-1248 (PCB-1248)
- 8905 Aroclor-1254 (PCB-1254)
- 8910 Aroclor-1260 (PCB-1260)
- 184 Aroclor-1262 (PCB-1262)
- 103 Aroclor-1268 (PCB-1268)

EPA 8260B	10184802	Volatile Organic Compounds by purge and trap GC/MS
Analyte Code	Analyte	
5105	1,1,1,2-Tetrachloroethane	
5160	1,1,1-Trichloroethane	
5110	1,1,2,2-Tetrachloroethane	
5195	1,1,2-Trichloro-1,2,2-trifluoroethane	
5165	1,1,2-Trichloroethane	
4630	1,1-Dichloroethane	
4640	1,1-Dichloroethylene	
4670	1,1-Dichloropropene	
5150	1,2,3-Trichlorobenzene	
5180	1,2,3-Trichloropropane	
5155	1,2,4-Trichlorobenzene	
5210	1,2,4-Trimethylbenzene	
4570	1,2-Dibromo-3-chloropropane (DBCP)	
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)	
4610	1,2-Dichlorobenzene	
4635	1,2-Dichloroethane	
4655	1,2-Dichloropropane	
5215	1,3,5-Trimethylbenzene	
4615	1,3-Dichlorobenzene	
4660	1,3-Dichloropropane	
4620	1,4-Dichlorobenzene	
4665	2,2-Dichloropropane	
4410	2-Butanone (Methyl ethyl ketone, MEK)	
4500	2-Chloroethyl vinyl ether	
4535	2-Chlorotoluene	
4860	2-Hexanone	
4540	4-Chlorotoluene	
4995	4-Methyl-2-pentanone (MIBK)	
4315	Acetone	
4325	Acrolein (Propenal)	
4340	Acrylonitrile	
4375	Benzene	
4385	Bromobenzene	
4390	Bromochloromethane	
4395	Bromodichloromethane	
255	Bromoethane (Ethyl Bromide)	
4400	Bromoform	
4950	Bromomethane (Methyl bromide)	
4450	Carbon disulfide	
4455	Carbon tetrachloride	
4475	Chlorobenzene	
4485	Chloroethane	
4505	Chloroform	



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Cusotmers: Please verify the current accreditation standing with ORELAP.

4960	Chloromethane (Methyl chloride)
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4560	Cyclohexanone
4575	Dibromochloromethane
4595	Dibromomethane
4625	Dichlorodifluoromethane
4765	Ethylbenzene
4835	Hexachlorobutadiene
4870	Iodomethane (Methyl iodide)
4900	Isopropylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
4910	p-Isopropyltoluene
4440	sec-Butylbenzene
5100	Styrene
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
4605	trans-1,4-Dichloro-2-butene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane
5225	Vinyl acetate
5235	Vinyl chloride

EPA 8270D 10186002 SemiVolatile Organic compounds by GC/MS

<u>Analyte Code</u>	<u>Analyte</u>
6715	1,2,4,5-Tetrachlorobenzene
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
6380	1-Methylnaphthalene
183	2,2'-Oxybis(1-chloropropane)
6735	2,3,4,6-Tetrachlorophenol
6740	2,3,5,6-Tetrachlorophenol
6835	2,4,5-Trichlorophenol
6840	2,4,6-Trichlorophenol
6000	2,4-Dichlorophenol
6130	2,4-Dimethylphenol
6175	2,4-Dinitrophenol
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
5795	2-Chloronaphthalene
5800	2-Chlorophenol

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Cusotmers: Please verify the current accreditation standing with ORELAP.

6385	2-Methylnaphthalene
6400	2-Methylphenol (o-Cresol)
6460	2-Nitroaniline
6490	2-Nitrophenol
5945	3,3'-Dichlorobenzidine
6465	3-Nitroaniline
7365	4,4'-DDT
6140	4,6-Dinitro-2-methylphenol
5660	4-Bromophenyl phenyl ether
5700	4-Chloro-3-methylphenol
5745	4-Chloroaniline
5825	4-Chlorophenyl phenylether
6410	4-Methylphenol (p-Cresol)
6470	4-Nitroaniline
6500	4-Nitrophenol
6115	7,12-Dimethylbenz[a]anthracene
5500	Acenaphthene
5505	Acenaphthylene
5510	Acetophenone
5545	Aniline
5555	Anthracene
123	Azobenzene
5595	Benzidine
5575	Benzo[a]anthracene
5580	Benzo[a]pyrene
5585	Benzo[b]fluoranthene
5590	Benzo[g,h,i]perylene
5600	Benzo[k]fluoranthene
5610	Benzoic acid
5630	Benzyl alcohol
5640	Biphenyl
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl)ether
6255	bis(2-Ethylhexyl)phthalate (DEHP)
5670	Butyl benzyl phthalate
5680	Carbazole
5855	Chrysene
5895	Dibenz[a,h]anthracene
5905	Dibenzofuran
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
3800	Ethyl parathion
6265	Fluoranthene
6270	Fluorene
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
6285	Hexachlorocyclopentadiene
4840	Hexachloroethane
6315	Indeno[1,2,3-cd]pyrene

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Cusotmers: Please verify the current accreditation standing with ORELAP.

6320	Isophorone
7825	Methyl parathion (Parathion, methyl)
5005	Naphthalene
5015	Nitrobenzene
6530	n-Nitrosodimethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine
6605	Pentachlorophenol
6615	Phenanthrene
6625	Phenol
6665	Pyrene
6670	Quinoline

EPA 9010C	686	Total and Amenable Cyanide by Distillation and UV-Vis
<u>Analyte Code</u>	<u>Analyte</u>	
1510	Amenable cyanide	
1645	Total cyanide	

**APPENDIX C**  
**LABORATORY QA MANUAL**

**Analytical  
Resources Inc.  
Quality  
Assurance  
Plan**



## Quality Assurance Plan

Analytical Resources, Inc.  
4611 S. 134<sup>th</sup> Place, Suite 100  
Tukwila, WA 98168-3240

Revision 12-007  
4/11/06

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No web access? Phone 206-695-6200

<http://arilabs.com/cgi-bin/rcheck.cgi?f=LQAP&r=R12007>

This Quality Assurance Plan is approved and authorized for release by:

Mark Weidner  
Laboratory Director

David Mitchell  
Quality Assurance Manager



# Quality Assurance Plan

## Analytical Resources Inc.

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## **SECTION 1: INTRODUCTION**

### **Quality Assurance Policy and Objectives**

Analytical Resources, Inc. (ARI) is dedicated to providing accurate and reliable data in a timely and cost effective manner. The management of ARI is committed to analytical excellence and will provide the facilities and a professional environment to achieve this goal. The quality assurance program detailed in this document sets forth the policies and procedures that are followed by ARI to ensure that all reported results are both legally defensible and of the highest quality.

To ensure that data quality goals are achieved, the following characteristics must be considered:

#### **Precision, Bias and Accuracy**

For all analyses, there is a degree of uncertainty or error in the measurement process. This measurement error is generally one of two types: random error (precision) or systematic error (bias). Precision is a measure of agreement between replicate measurements. Bias is considered to be the difference between the expected value and the true value for a measurement or series of measurements. Accuracy is a determination of how closely a measurement is to the expected value. Both precision and bias are considered when determining the accuracy of measurements. Precision, bias and accuracy are evaluated through the use of method guidelines, and project and laboratory control limits.

#### **Representativeness**

Representativeness is an indicator of how closely one sample aliquot resembles another aliquot from the same bulk source or sample site. Sample representativeness is more easily obtained for particulate-free water samples than for solid samples or viscous liquids. Representativeness is an important consideration in achieving other data quality objectives.

#### **Completeness**

Completeness is an indicator of the number of valid (useable) data points compared with the overall number of data points obtained. Valid data are normally obtained when sample collection and analysis is performed in accordance with specified methods and procedures. Completeness is often expressed as a percentage: the higher the number of valid data points, the higher the overall completeness percentage. Conversely, fewer valid data points will result in an overall lower percentage of completeness. Project specifications will dictate the required level of completeness.





### **Comparability**

Comparability is an indicator of how confidently one data set can be compared with another, as well as the consistency between data sets. Stable analytical conditions and adherence to standard procedures, combined with high levels of accuracy; help ensure that results obtained over a period of time will be comparable.

### **Timeliness**

To ensure that the most accurate results possible are obtained, samples must be processed within specified time periods. Analytical holding times have been established to allow sufficient time for sample processing without compromising sample integrity. It is important that, while meeting timeliness requirements, other data quality objectives are still considered and met.

### **Documentation**

Complete and accurate documentation is essential for verifying the integrity of analytical results. Achievement of other quality objectives cannot be used to substantiate data quality without full documentation of the analytical process. Documentation must be concise and readily available for subsequent review.

The quality assurance program at ARI has been developed to ensure that the specified data quality objectives are met for all reported results and the highest degree of completeness possible is achieved.

## **1.2 Ethics Policy on Data Quality and Confidentiality**

To ensure that data quality or confidentiality is not compromised, ARI has established the following policy on corporate ethics. Following are steps that must be taken when the quality or confidentiality of data is suspected or known to be compromised. This policy applies to all ARI employees at every organizational level.

### **General**

ARI's corporate commitment to integrity and honesty in the workplace is clearly stated in the ARI Employee's Handbook, under "Standards of Conduct". The Standards of Conduct statement is attached as Appendix O. The ARI commitment to excellence in data quality extends to and includes all aspects of data production, review and reporting.

Any attempt by management or any employee to compromise this commitment presents a case for serious disciplinary action. Any indications or allegations of waste, fraud or abuse will be rigorously investigated by ARI management, with the penalties for verified cases to be employment termination, and if appropriate, prosecution. In addition to these steps, any such



charges related to data generated for the federal government will also be reported to the Inspector General of the appropriate department.

### **Circumstances**

All ARI employees will immediately report to management any information concerning the misrepresentation or possible misrepresentation of analytical data (or any associated components).

Misrepresentation of data includes (but is not limited to) the following:

*Altering an instrument, computer or clock to falsify time or output*

*Altering the content of a logbook or data sheet in order to misrepresent data*

*Falsifying analyst identity*

*Changing documents with correction fluid with the intent of falsifying information*

*Preparing or submitting counterfeit data packages or reports*

*Unauthorized release (either written or verbal) of confidential data*

*Illegal calibration techniques (peak shaving, fraudulent integrator parameters)*

*Any attempt to misrepresent data or events as they actually occur in the course of data production or reporting*

### **Responsibilities**

It is the responsibility of all ARI employees to report any situation which may be adverse to data quality or confidentiality, or which may impact the final data quality. All ARI employees have the obligation to discuss known or suspected violations of this policy with laboratory management, who in turn are obliged to inform the ARI Laboratory Manager. If a satisfactory resolution is not obtained or is not possible at laboratory level, all ARI employees have the right and responsibility to discuss the matter directly with the ARI Laboratory Manager.

It is the responsibility of the ARI Laboratory Manager to promptly investigate any reports of known or suspected violations. The ARI Laboratory Manager has the authority and responsibility to resolve all known or potential violations of the policy.

It is the responsibility of ARI management to provide all of its employees with the facilities, equipment, and training to achieve the quality goals stated in the policy. It is the responsibility of ARI to provide our clients with data of known and documented quality.



## **Documentation**

To reaffirm an awareness of and commitment to the highest standards of data quality, excellence, and integrity, all employees are required to sign the following “Commitment to Excellence in Data Quality” statement:

“As an ARI employee, I have the right and responsibility to report any situation which may be adverse to quality or which may impact the final quality or integrity of data produced for our clients.”

“I will report immediately to management any information concerning the misrepresentation or possible misrepresentation of analytical data (or any of its associated components). Examples of this include (but are not limited to): alteration of an instrument computer or clock, alteration of the contents of logbooks and/or data sheets in order to misrepresent data, misrepresentation of analyst identity, intentional falsification of documents with correction fluid (“white-out”), preparation and submittal of counterfeit data packages, use of illegal calibration techniques (peak shaving, use of fraudulent integrator parameters, etc.), or any attempt to misrepresent data or events as they actually occur in the course of an analysis.”

“I will likewise alert management of any situation or activity which may be adverse to the confidentiality of clients’ data.”

“I will not knowingly participate in any such activity, nor fail to report such activities of which I may become aware. I understand that any voluntary participation on my part in such activities may result in the termination of my employment, and possible legal prosecution.”

“Where circumstances permit, I will report any actual or suspected violations of this policy to my lab or section supervisor. If a satisfactory resolution is not obtained or is not possible at that level, I have the right and obligation to discuss the matter directly with the ARI Laboratory Manager.”

## **Confidentiality**

All information related to client projects, such as client work plans, documentation and analytical data will be considered confidential. This information will be released only to the client or an authorized representative. Should an outside agency request information related to a client project, the client will be contacted for approval prior to releasing any information.



Some programs or contractual agreements (such as the USEPA Contract Laboratory Program) may have specific requirements for protecting a client's confidentiality. Project Managers will be responsible for strict control of access to any such confidential information or documentation. All data generated from the analysis of confidential samples will also be considered confidential.



## **SECTION 2.0: QA MANAGEMENT AND RESPONSIBILITIES**

The principal tenet of the Quality Assurance Program at Analytical Resources Inc. (ARI) is that every employee knows she/he is a vital component of the program, and holds a responsibility to produce high-quality, defensible data in a timely manner. While production of quality data is a global philosophy, held by the entire laboratory, each section is responsible for ensuring that the data produced within that section meets the required quality objectives.

### **2.1 Overall Structure**

The Board of Directors shall direct ARI's QA Policy and shall determine the Philosophy of the QA Program. It shall be the responsibility of the Laboratory Director to translate this policy into practical procedures with respect to the business plan developed for ARI, and direct the Laboratory Manager and Section Managers regarding the incorporation of these procedures into daily laboratory activities.

The Laboratory Manager is responsible for coordination of laboratory activities to result in an integrated approach to quality data production. The Laboratory Manager will coordinate Client Services, Laboratory Section Management, Computer Services, and Data Services to ensure that project requirements and data quality objectives are met.

The Laboratory Section Managers and Supervisors shall hold the final authority in decisions concerning implementation of QA policy, with the contributions of the Laboratory Director, Laboratory Manager, QA Manager and Project Managers. Section Managers and Section Supervisors shall instruct employees in the proper employment of QA policies.

Each Section Supervisor will ensure that analyses are completed within required holding times, that data is submitted within required submission times, and all analyses are performed according to the current Standard Operating Procedures (SOPs). They will ensure that any client modifications or QA issues are well documented for each sample set and that all required documents are complete when submitted with each data set.

The analytical staff shall execute all methods following QA policies, and will write SOPs reflecting the methods exactly as performed. These SOPs will be reviewed for compliance by



Section Managers and the Laboratory Director, and once approved will be submitted to the Quality Assurance Program Manager (QAPM).

The QAPM will be responsible for controlling Company SOPs and other internal documents, overseeing the scheduling and completion of detection limit studies. The QAPM will coordinate the production of control charts and distribution of control limit data to all laboratory sections. The QAPM will administer the blind QA proficiency tests and performance samples as described in the QA Program. The QAPM will verify that QA policies and procedures are followed through out ARI.

Data reviewers will be responsible for ensuring that all samples have been analyzed by the approved and requested methods, that data calculations are performed correctly, and that analyses meet the Data Quality Objectives of the client. They shall also be responsible for ensuring that the documentation from each laboratory section is intact and complete.

Computer Services is responsible for ensuring that the Laboratory Information Management System (LIMS) correctly reflects the preparations and analyses performed and that the LIMS is updated with the current SOP, MDL, RL and QL data as submitted from the QAPM. Computer Services personnel are also responsible for ensuring that all electronic deliverables for clients are formatted correctly as requested by the Project Managers and that this data matches the hardcopy deliverables submitted.

Client Services (Project Management, Sample Receiving), shall be responsible for ensuring that the laboratories understand and can meet project specific analytical requirements and DQO.

## **2.2 Hierarchical Responsibilities**

### **Technical Director**

*It shall be the responsibility of the Laboratory Director to translate QA policy into practical procedures with respect to ARI's business plan, and to direct the Laboratory Manager and Section Managers in the implementation of these procedures in daily laboratory activities.*

The Director shall interpret overall QA Policy, and determine the broad practicality of policies based on methodologies, technological advances, and the current environmental market. It



shall be the interpretation of these policies that will, in turn, direct the growth ARI, the addition or withdrawal of methods to ARI's repertoire, and ARI's marketing focus.

At a minimum of once a year the Technical Director shall include on the agenda of the Board of Directors meeting a discussion of ARI's QA Policy. This discussion will include the reputation of ARI for producing quality analyses, the affect of QA policies on turn-around time, competitive edge and cost-of-analysis, needs for stricter or more flexible policies, and the response of employees to the QA policies in place at that time.

At a minimum of once every six the Director shall attend management meetings, which include on the agenda the subject 'QA Program'. This format will allow for the dissemination of information on any QA issues addressed in the laboratory or by the Board of Directors. Management shall also use these meetings to discuss requirements of clients that are not met by ARI's present QA Program, and the appropriate response to these requirements.

The Technical Director may be required to act as a technical advisor at any impromptu meetings called by management to address QA issues that cannot be immediately resolved within a laboratory section.

It shall also be the Director's authority and responsibility to hold final review approval for all SOPs of ARI. Once an SOP has been updated and reviewed by the laboratory section, it shall go through the Section and Laboratory Managers for approval, and then to the Laboratory Director for final approval before the SOP is released.

### **Laboratory Manager**

*The Laboratory Manager is responsible for coordination of laboratory activities to result in an integrated approach to quality data production. It shall be the Laboratory Manager's responsibility to coordinate Client Services, Laboratory Management, Computer Services, and Data Services to ensure that QA Program requirements and data quality objectives are met.*

The Laboratory Manager is required to attend all management meetings, at which the QA Program will be an agenda item. Management shall use these meetings to discuss requirements of clients that are not met by ARI's present QA Program, the appropriate response to these requirements, and dissemination of information on any QA issues addressed in the laboratory or by the Board of Directors.



It is the responsibility of the Laboratory Manager, along with the QA Manager, Laboratory Director, Section Managers and Client Services, to determine in which QA Proficiency Programs the Laboratory will participate, and those accreditations that ARI will pursue. It is the responsibility of the Laboratory Manager, with the Section Managers, to ensure that all laboratory sections perform the tasks required by the QA Manager to pursue each accreditation or to complete a scheduled audit.

The Laboratory Manager has the responsibility of balancing client requests and requirements with the QA policies of ARI. It is the Laboratory Manager's task to evaluate a client's Data Quality Objectives (submitted through Client Services), and with the Section Managers to determine the feasibility of laboratory performance. Feasibility will be based on the quality objectives requested, current QA Manual, present workload (in-house and scheduled/pending), the technology in place, and staffing levels available. Current workload in-house will be evaluated using reports from Computer Services, and scheduled/pending workload will be evaluated using written and verbal input from Client Services.

The Laboratory Manager has the authority to direct Client Services to discontinue the bidding/contracting process for a new project, refuse samples, or to re-schedule projects based on Data Quality Objectives or current workload. The Laboratory Manager also shall evaluate staffing and equipment needs based on information from the Section Managers and Client Services and may elect to meet new project requirements by increasing staffing levels or purchasing additional equipment.

The Laboratory Manager serves as a senior-level technical reference for all laboratory activities, and as such will be brought in to advise on out-of-control events and trends, corrective actions, and/or other QA issues that require his/her expertise.

### **Laboratory Section Managers**

*The Section Managers shall hold the final authority in decisions concerning implementation of QA policy, with the contributions of the Laboratory Director, Laboratory Manager, QAPM and Project Managers. Section Managers are responsible for correcting out of control events within their respective laboratories. Section Managers and supervisors shall instruct employees in the proper employment of QA Policies.*





Laboratory Sections Managers shall have the final authority in decisions concerning QA policy. It is their expertise that will determine the final acceptable format of each method SOP, as they are the best resource to integrate methods into ARI's philosophy.

Laboratory Section Managers are responsible for completing or delegating updates of laboratory procedures and quality assurance manual sections as scheduled by the QA Manager.

The Section Managers are best able to determine capacity of the Laboratory Sections. To ensure that analyses are completed within required hold times, the Section Managers will give Supervisors the authority to balance employee workloads and modify employee work schedules. It is the Section Manager's responsibility to take reports from supervisors and work with the Laboratory Manager to increase staffing levels or reject samples as needed. It is the Section Manager's responsibility to work with the Laboratory Manager and the section supervisor and analysts to ensure that sample capacity does not affect the quality of data generated from that laboratory section.

It is the responsibility of the Laboratory Section Managers, along with the QA Manager, Laboratory Director, Laboratory Manager and Client Services, to determine in which QA Proficiency Programs the Laboratory will participate, and which accreditation processes ARI will pursue. It is the responsibility of the Section Managers, with the Section Supervisors, to ensure that all laboratory sections perform the tasks required by the QA Manager to pursue each accreditation or to complete a scheduled audit.

The Section Manager will be responsible for reviewing training records of analysts produced by the Section Supervisor. Training shall be the responsibility of the Section Supervisor, but it is the responsibility of the Section Manager to oversee this training.

It is the Section Managers' responsibility to work with the Section Supervisor and Project Manager to assure that Project Requirements are achievable and valid for the given methods. At times, ARI's clients have requests or requirements for methods that are 1) not the method of choice in the laboratory, 2) not presently performed by the laboratory, or 3) unachievable by the instrumentation used in the laboratory. It is the responsibility of the Section Supervisor,



Section Manager and Project Manager to work with the client to resolve these issues before samples are accepted.

Clients may also request modifications to the methods that must be approved by the Section Supervisor, the Section Manager and the QAPM. These modifications must be thoroughly documented and all pertinent information on modifications must be conveyed to the analysts, sample preparation sections, sample receiving, and computer services, as needed for implementation.

The Section Manager is responsible for resolution of out-of-control events that have not or cannot be resolved by the analysts or Section Supervisor.

The Section Manager has the authority to re-classify analysts or require additional training of analysts based on their performance.

### **Section Supervisors**

*It is the responsibility of each section Supervisor to ensure that analyses are completed following the most current version of ARI's SOP, within required holding and turn around times, and assure that analyses meet the Data Quality Objectives of each project. They will ensure that any client modifications or QA issues are well documented for each sample set, and that all documentation is complete when submitted with each data set.*

To ensure that analyses are completed within required hold times, the Supervisors have the authority to balance employee workloads and modify employee work schedules. The Section Supervisors, with the input of the Section Manager, have the authority to request overtime from employees should the workload warrant the additional effort, or to modify employee schedules to extend the operating hours of the laboratory section.

The Section Supervisors shall oversee the day-to-day section operations, using LIMS printouts and verbal or written workload estimates and requests from Project Managers to adjust section efforts as needed. It is also the Section Supervisors' responsibility to inform management (Section Manager, Data Review, and Project Managers), when capacities are limited, so that the appropriate adjustments can be made to reduce workloads or increase laboratory capacities. At no time should sample capacity be allowed to affect the quality of data generated from any laboratory section.



It is the Section Supervisor's responsibility to assure that employees have the proper training for their positions. This training will include training in the methods, use of the LIMS system if applicable, training in correct documentation procedures, and all information necessary for adherence to the ARI QA Program. The Supervisor shall either perform the training personally, or designate the trainer for given methods or procedures. It is the Supervisor's responsibility to test each employee for each method or procedure, and to thoroughly document each employee's advances and current capabilities. The Supervisor shall have the authority to require further training or supervision for any employee, and shall be the authority to approve each employee for working without supervision. There will be a training record for each employee. These will be kept in the laboratory section; copies will be submitted to the QA Manager for record keeping.

It is the Supervisor's responsibility to work with the Section Manager and Project Manager to ensure that Project Requirements are achievable and valid for the given methods. At times clients have requests and/or requirements for methods that are 1) not the method of choice in the laboratory, 2) not presently part of the method as performed by the laboratory, or 3) unachievable by the instruments used in the laboratory. It is the responsibility of the Supervisor, Section Manager and Project Manager to work with the client to resolve these issues before samples are accepted.

It is the responsibility of the Section Supervisor to ensure that each analyst reads and understands all requirements submitted with each sample set, including those for any special analyte, calibration, or data deliverable. It is the Section Supervisor's responsibility to clarify any issues, with the input of the Section Manager and the Project Manager for the client.

Clients also at times will request modifications to methods, which must be approved by the Supervisor and Section Manager. These modifications must be thoroughly documented and all pertinent information on modifications must be conveyed to the analysts, sample preparation sections, sample receiving, and computer services as needed for implementation.

It is the Supervisor's responsibility to ensure that each employee understands the requirements of all projects they work with. This may necessitate section meetings or project-



specific cross-section teams to work with Project Managers for large, specialty projects to ensure that everyone has the same understanding of project requirements.

The Supervisor is responsible for resolution of out-of-control events that have not or cannot be resolved by the analysts, and for ensuring that the analysts complete all documentation. If the Supervisor and laboratory section analysts cannot resolve the issues in a timely manner, the Supervisor's will request the assistance of laboratory management to bring the section into compliance. The Supervisor will also inform Project Management and his/her Section Manager of possible delays, and inform Data Review of possible time constraints they may face in preparation of data submissions from the lab section.

The Section Supervisors shall have the authority, usually in consultation with Laboratory or Project Management to use professional judgment in requiring samples be re-prepared, and shall determine which analysts have the authority to require re-preparation of samples.

It is the responsibility of the Section Supervisor to inform the QAPM, Section Manager and the Computer Services section of any changes in methodologies that will require revision of SOPs, MDLs, Control Limits or the LIMS programming. This includes changes in spiking compounds, spiking levels, preparation methods and analytical methods.

### **Analysts**

*The analytical staff shall execute all methods following QA Policies, and will write SOPs reflecting the methods exactly as performed. These SOPs will be reviewed for compliance by Section Managers, the Laboratory Manager, and the Laboratory Director, and once approved will be submitted to the QA Manager.*

The analysts are responsible for following the current SOPs (with project-specific modifications if required) in preparing and analyzing client samples and quality control samples to meet the project specific Data Quality Objectives. It is the analyst's responsibility to ensure that he/she understands all requirements of a project before proceeding with sample preparation or analysis.

Analysts are responsible for working with the Supervisor to ensure that all sample preparations and analyses are performed within required holding times and required turn-around times, and that all documentation is completed in a timely fashion. It is each analyst's responsibility to bring any recurrent or anticipated problems to the attention of laboratory management.



It is each analyst's responsibility to correct his/her own errors, to document corrective actions thoroughly, to perform peer review, and to ensure that fellow employees within the section follow documentation procedures.

The Section Supervisor may give lead analysts responsibility for training and evaluation of new staff members. This training will include instruction in the methods, use of the LIMS system if applicable, correct documentation procedures, and all information necessary for adherence to the ARI QA Program. Analysts will be responsible for maintaining all instruments and equipment in optimum operating condition and documenting this maintenance as required by the QA Program.

It is the responsibility of each analyst to request the assistance of Supervisors or Managers in resolving out-of-control situations that cannot be corrected in a timely manner, and to perform the documentation of all corrective action activities.

### **Quality Assurance Program Manager (QAPM)**

*The QAPM will be responsible for controlling Company SOPs and other internal documents. The QAPM will oversee the scheduling and completion of detection limit studies and control charts. The QAPM will administer the training program, analyst's proficiency documentation and performance evaluation analyses as described in the QA Program. The QAPM will verify that QA policies and procedures are followed at all levels in the Company. The QAPM will produce a "Quality Assurance report to Management" each calendar year.*

The QAPM is responsible for the oversight of the QA Program as defined by the Board of Directors and interpreted by the Laboratory Director and Laboratory Managers.

Part of this oversight will be monitoring of the QA Program through submission of performance evaluation samples, blind QA samples and double-blind QA samples. It is the responsibility of the QAPM, along with the Laboratory Manager, Laboratory Director, Section Managers and Client Services, to determine in which QA Proficiency Programs the Laboratory will participate. The QAPM will be responsible for submitting these samples to the laboratory for analysis, overseeing submission of the results to the appropriate agencies, and for control of documented proficiency results.

The QAPM will be responsible for scheduling laboratory section SOP and procedural reviews and revisions, and section updates of the Quality Assurance Manual. It is the responsibility of



the QAPM to work with each Section Manager to attempt to stagger these review schedules across the year within each laboratory section. The QAPM will also be responsible for maintaining document control of all SOPs, bench sheets, logbooks, and other forms used within the laboratory.

All laboratory sections, on an annual basis, will perform detection limit studies for each method used within each section. It is the responsibility of the QAPM to schedule, review, compile, and distribute the results of these studies.

The QAPM is responsible for evaluation of the laboratories' adherence to defined protocols through periodic audits of completed projects and of the laboratory facilities. Following the audit schedule (Appendix K), the QA Manager will perform the scheduled audit and prepare an evaluation that will be submitted to the Board of Directors in the Annual QA Report to Management.

The QAPM will be responsible for evaluation of outside accreditation requested by Client Services. The QA Manager will deliberate with the Laboratory Managers and Laboratory Director on the feasibility of pursuing accreditation based on the scope of the accreditation, the effort required to pursue accreditation and the scope of work that might become available once the accreditation is obtained. If a decision is made to pursue an accreditation, it is the responsibility of the QAPM to coordinate laboratory efforts towards the accreditation.

The QAPM will produce an annual "Quality Assurance Report to Management" to be distributed to ARI management personnel as described in Section 13 of this LQAP.

The QAPM will serve as a resource for quality-related issues for all Laboratory Sections, and will serve management in an advisory capacity.

The QAPM will have documented training in elementary statistics and Quality Systems theory.

### **Data Reviewers**

*Data reviewers will be responsible for ensuring that all samples have been analyzed by the approved and requested methods, that data calculations are performed correctly, and that analyses meet the Data Quality Objectives of the client. They shall also be responsible for ensuring that the documentation from each laboratory section is intact and complete.*



Data reviewers shall ensure that all samples are analyzed according to approved methods by reviewing the data released by each laboratory section. The data will be evaluated for compliance with all Data Quality Objectives as defined in the method SOP or in the project-specific quality assurance plan, including instrument tuning and calibration, holding time, spiking level, and spiking recovery criteria. Data reviewers will also verify 100% of manual calculations, spot check computer calculations, check electronic data for correct sample matching, and do a 100% check on any manually entered data. Analytical parameters, which have concentration interdependence, will be evaluated in relationship to each other.

Final reports generated will be evaluated to ensure that laboratories are using the current detection limit/reporting limit values and the current control limits. Data will be checked to ensure that all QA issues are addressed and fully documented. Reviewers are responsible for working with Laboratory Supervisors, Laboratory Managers and Project Managers when out-of-control events are incompletely documented, or if data is found to not meet Data Quality Objectives of a project without documentation.

It is the responsibility of data reviewers to work with Computer Services to ensure that the LIMS is updated to the current limits and methods used within the laboratory.

### **Computer Services**

*Computer Services is responsible for ensuring that the LIMS correctly reflects the preparations and analyses performed and that the LIMS is updated to include the current SOP, MDL, RL and QL data, as submitted by the QA Manager. Computer Services personnel are also responsible for ensuring that all electronic deliverables for clients are formatted correctly as requested by the Project Managers and that electronic data matches the hardcopy deliverables submitted.*

It is the responsibility of the Computer Services Manager to update, or to designate the task of updating, the LIMS as determined by Laboratory Management, including adjustment to current MDL/RL data, additions of analytes to methods, changes in method designations or changes in calculations for methodologies.

Computer Services will be responsible for generating the work list scripts required to allow analysts to enter data into the LIMS, and for generating the report scripts that produce final hardcopy or electronic reports for clients.



Computer Services Management and personnel are also responsible for generation and review of electronic data deliverables (EDD), as requested by clients through Project Management. Computer Services personnel will review the EDD for compliance with the Software Quality Assurance SOP before it is released to the client.

Computer Services will be responsible for informing laboratory Section Managers and Project Managers of any discrepancies found between the EDD and the hardcopy, and for following up on corrections to hardcopy and EDD as required.

### **Client Services**

*Client Services (CS) (Project Managers, Sample Receiving, and Sales Management) personnel are the primary interface between ARI's clients and the laboratory sections. CS staff shall be responsible, with the assistance of the Section Managers and Supervisors, for ensuring that the laboratories understand and can meet the Data Quality Goals and Requirements of each Project before committing laboratory services to the project. CS will monitor the quality of sample processing after they are received.*

Client Services Management and Project Managers shall ensure that the laboratories can meet the data quality objectives for a project. The Project Managers are responsible for knowing the capabilities of the laboratory, in order to develop project proposals or accept samples without consultation with laboratory management. It is the responsibility of Client Services to consult with the Laboratory Manager and Section Managers, or supervisors designated by Management, when data quality goals are not included in standard Company policies. Clients may, at times, request modifications to methods that must be approved by the Supervisor and Section Manager. These modifications must be thoroughly documented and all pertinent information on modifications must be conveyed to the analysts, sample preparation sections, sample receiving, and computer services as needed for verification of feasibility. Laboratory Management may determine that a project should not be pursued based on the specific Data Quality Objectives and on current or projected laboratory capacity.

Project Managers shall be responsible for ensuring that project requirements and analytical requests are submitted correctly to all laboratory sections. Once samples have been logged into the laboratory, it is the responsibility of the Project Managers to ensure that all information is available to the laboratories concerning the Data Quality Objectives and deliverables





requirements. It is also the responsibility of the Project Managers to convey changes in client requirements to the laboratories and ensure that all paperwork reflects the changes if necessary.

It is the responsibility of Project Managers and Client Services Management to assure that specific EDD formats are submitted to Computer Services and approved as feasible before contracting with a client to provide the EDD.

It is the responsibility of Project Managers to notify clients of out-of-control events, “problem” samples, or anticipated turn-around time delays, as conveyed to them by Laboratory Management. It is also the responsibility of Project Management to work with Laboratory Management in setting priorities during times of heavy sample workloads.

Project Managers shall be responsible for coordinating data submissions and compiling hardcopy data for final submission to the client. This involves conducting a fourth level data review, from which any data which is found to contain errors that were not found earlier in the review process is returned to the Data Reviewer for correction and/or corrective action. The Project Manager will be responsible for compiling all analyst notes into a project narrative. This will include discussion of any sample receipt discrepancies, sample preparation and analysis difficulties or non-compliance, and any corrective actions that may have been required during processing. It will also discuss quality control analyses and results if applicable to the sample set.

Project Managers shall work with Laboratory Management in determination of the direction of growth for ARI, as the Project Managers are best able to define the analytical needs of clients based on new technologies and new environmental regulations.

## SECTION 3: PERSONNEL QUALIFICATIONS AND TRAINING

The production of quality analytical data is dependent upon a laboratory staff with qualifications and training necessary to perform assigned tasks. All personnel employed by ARI will receive adequate training and instruction specific to their responsibilities. Prior to assigning a staff member full responsibility for performing a laboratory procedure, her/his skills will be evaluated and verified acceptable. It is the obligation of ARI's supervisors and managers to ensure that personnel are qualified to successfully perform all assigned duties.

ARI's training program is described in SOP 1017S (*Training and Demonstration of Proficiency*). The procedures described in this SOP assure that all ARI employees are proficient at the tasks required to produce quality analytical data. The SOP also provides for periodic review of each employees training and proficiency status, which may indicate any need for additional or remedial training. All training and review procedures are documented as described in the SOP. Appendix B of this document includes specific requirements of the training program and examples of the forms used to document training.

Basic elements of ARI's training program are:

1. All employees are required to read and document their knowledge of non-technical documents that describe general policies in place at ARI. These documents include ARI's *Employee Manual* and ARI's *Chemical Hygiene Plan*.
2. All employees are required to read and document their knowledge of ARI's *Laboratory Quality Assurance Plan* and quality assurance policies.
3. All new employees must attend a Quality Assurance Orientation during which ARI's general and specific requirements for the production of quality analytical data are emphasized. A typical orientation agenda is included in Appendix B.
4. All new employees will attend a Technical Orientation conducted by their laboratory supervisor or manager. The technical orientation is used to provide specific information about laboratory operation to the employee and to assess the new employee's education



and skill level. The section supervisor or manager is responsible for determining the level of training necessary for each staff member.

5. All employees will complete an 'on the job' training program designated by their supervisor. The training program will be laboratory, SOP and employee specific. Training programs follow the general outline provided in Appendix B. The training is incremental with each step documented in an employee Training File. While an analyst is in the training period, her/his supervisor or trainer must approve all analytical work. Upon completion of the training program the employee is considered proficient and may perform without supervision the SOPs listed in the training program.
6. The proficiency of each employee performing a given laboratory SOP will be continually monitored and documented as described SOP 1017S. An employee must continually generate data that meets all of ARI's published acceptance criteria for a given SOP to be considered proficient. Unacceptable results or insufficient number of analyses performed in a calendar quarter will result in revocation of proficiency. This will result in a remedial training program.
7. Periodically, as described in SOP 1017S, internal and/or external Performance Evaluation (PE) samples will be used to document staff competency. Technicians and analysts will participate in the preparation and analysis of blind samples for all methods they routinely perform. Results of these blind samples will be evaluated to verify staff proficiency. Staff members associated with acceptable performance evaluation samples will be considered proficient for those methods. Conversely, unacceptable performance evaluation sample results may signal the need for additional or remedial training.
8. A training file is established for each technical employee. The file will document an employee's experience, training and proficiency. The training file will document each specific PE sample analysis performed by an analyst. Either an employee's supervisor or Quality Assurance Program personnel will update the training file. The training file will be maintained in the employee's laboratory as outlined in SOP 1017S.



## SECTION 4: FACILITIES AND EQUIPMENT

### 4.1 Facilities

ARI's facilities have been designed to allow for efficient sample processing and analysis while maintaining consideration for the health and safety of the staff. The facility accommodates the following operations:

*Sample receipt and storage*  
*Sample container preparation and shipment*  
*Sample preparation and analysis (organic and inorganic)*  
*Project planning and management*  
*Quality assurance*  
*Data review and report generation*  
*Computer programming and operations*  
*Records storage*  
*Instrument spare parts storage*  
*Short-term hazardous waste storage*

A detailed description of ARI's facilities is included as Appendix C.

### 4.2 Security

#### Facilities

To ensure that security at ARI is maintained, access to the facilities is limited to employees and escorted visitors. Upon arrival, ARI visitors are required to register at the reception desk, and must sign out prior to leaving. Visitors will be escorted at all times. A receptionist constantly monitors the main entrance. Other laboratory entrances remain closed at all times and can only be opened from the outside by key. Key access to the facility is controlled; keys are issued on a limited basis depending on access needs.

As a result of controlled access and a monitored alarm system, the entire facility is considered a secure area. This eliminates the need for locked sample storage refrigerators, data storage areas or file cabinets.

#### Data Access

The Computer Services Manager controls security of, and access to, electronic data on the LIMS. Security measures are required to ensure data integrity, but must not be so restrictive



as to prevent data accessibility. The security measures taken at ARI are to prevent intentional intrusion by outside parties. These measures include building security, limited computer system access, password systems, encryption, firewalls and the use of virus protection programs. ARI's Intranet is protected from outside tampering by a proxy server (firewall) connection to the Internet.

## **LIMS - System Security**

### **Building/Computer Room Security**

Access to the building is restricted to employees, vendors with security passes, and escorted visitors. Room 203 contains the computer and main console for the LIMS system. This room is closed and locked at all times. Access to this room is limited to Computer Services personnel, escorted repair technicians, and escorted visitors. Only Computer Services personnel will be allowed access to the main console.

### **System Password Policy**

User name and password restrict access to the LIMS computer. Remote access to the LIMS server is not allowed.

### **Database Access Restrictions**

Interaction with the database is menu-controlled and allows the LIMS Manager to restrict access. Technicians may be given the ability to fill a limited number of work lists, with no authorization to distribute data. Some users may be given "read only" access to the database.

Users will be given access to the database only to complete tasks for those analyses for which they are responsible. No users are to be given access to the shell or command prompt unless 1) they have completed the appropriate training and 2) administrative access to the computer systems is required by their job function

## **4.3 Safety**

Ensuring that all sample processing and analysis procedures are performed under safe conditions is an important consideration at ARI. While safety is the responsibility of all staff members, ARI's Safety Committee meets monthly to review the safety activities of all laboratory sections and to ensure that all operations and equipment meet safety criteria. *The*

*Chemical Hygiene Plan* details those safety procedures and requirements that must be followed at ARI. *The Chemical Hygiene Plan* is reviewed annually and updated as needed to incorporate any changes to ARI's safety program.

## **4.4 Instrumentation and Support Equipment**

### **4.4.1 Instrumentation**

Generation of quality data is dependent upon instrumentation and support equipment that is in optimum operating condition. All instrumentation will be optimally maintained per method requirements and manufacturer recommendations. Preventative maintenance will be performed on a routine basis, with more frequent maintenance during periods of increased sample load or after analysis of highly contaminated samples. Preventative maintenance is instrument and analysis specific, and each maintenance logbook has been designed specifically for the instrument type. Required maintenance procedures are listed in analytical SOPs. Each maintenance logbook will detail the type and frequency of maintenance for that instrument. Each maintenance logbook is kept with the instrument. Non-routine maintenance and repairs will be performed as necessary. Spare parts are kept on hand when possible; necessary parts are ordered on an expedited basis to minimize downtime. All maintenance and repair activities will be documented in the appropriate logbooks.

Currently available Laboratory Instrumentation is detailed in Appendix C.

### **4.4.2 Support Equipment**

4.4.2.1 Thermometers - – All thermometers in use at ARI are traceable to an NIST standard and are calibrated or verified annually. The procedures are described in SOP 1020S.

4.4.2.2 Water Baths, Incubators and Ovens – The temperature of water baths and ovens currently used in the analytical process are monitored daily. Temperatures are recorded on temperature logs that are audited monthly by Quality Assurance personnel. Temperature controls on these devices are calibrated annually by an outside vendor. Calibration reports are filed in the QA Office.



4.4.2.3 Refrigerators and Freezers - Refrigerators and freezers are assigned an acceptable operating range of temperatures. The temperature is monitored daily. Corrective Action is required for all out of range temperatures.

4.4.2.4 Balances – The accuracy of all balances is verified prior to daily use with two Class S weights that bracket the normal weighting range of the balance. All analytical balances are professionally cleaned and calibrated annually by an outside contractor. Class S weights are calibrated every three years by an outside contractor. Calibration reports are filed in the QA Office.

4.4.2.5 pH Meters – pH meters are standardized prior to daily use with at least two standards, one at 4.0 and one at 7.0 pH units. The meters are checked prior to each use with a pH 7.0 buffer.

4.4.2.6 Variable Volume Pipettes – The accuracy of variable volume pipettes is verified monthly following the procedure in SOP 1015S.

4.4.2.7 Sample Containers – Upon client request ARI will supply sample containers for collection of field samples. All containers supplied for organic and trace metals analyses are certified pre-cleaned by the manufacturer. Containers for Conventional analyses are not pre-cleaned and are certified internally by ARI following the procedures in Appendices of ARI SOP 001S (Sample Receiving). The manufacturer's certification may be above ARI's reporting limit for some analyses. When this is the case ARI performs a Method Blank analysis using a container from a given lot and certifies that the lot is suitable for sample collection.

Container lot numbers are recorded when containers are sent to a client.

### **4.4.3 Chemical Standards and Reagents**

#### **4.4.3.1 Reagent Water Supply**

ARI maintains a centralized water purification system. The quality of the water produced is monitored and documented daily. Routine maintenance, out of control events and corrective actions are documented for each system in a logbook. All reagent / de-ionized water used within the laboratory meet or exceed ASTM Type II Standards. Water used in the Volatile Organic Laboratory is also filtered through activated charcoal to remove organic compounds. .

#### **4.4.3.2 Chemical Standards**

All quantitative standards used for calibration in an analytical process are traceable to a National Institute of Standards & Technology standard if one exists. Non-traceable standards are verified against traceable standards or through the analyses of Standard Reference Materials. A Certificate of Analysis is filed in the QA Section or laboratory for all quantitative standards. The source, date of receipt, required storage conditions and an expiration date will be documented for all standards. All containers used to store standards will be labeled with an expiration date. Receiving, storage and preparation of calibration standards is described in SOPs 526S (Metals Analysis), 620S (Conventional Analysis), 704S (Volatile Organic Analysis) and 1012S (GC and GC-MS Analyses).

#### **4.4.3.3 Chemical Reagents**

Many of the analytical processes in use at ARI require chemical reagents that are not directly used in the calibration process. These reagents are used to accomplish such tasks as analyte preservation, adjustment of pH, the forming of colorimetric indicators, etc. These reagents are purchased in a grade and purity sufficient for their intended use. The receipt of all reagents is recorded in the Chemical Receiving Logbook where a unique Inventory Number is assigned to each reagent. Each original reagent container is labeled with an Inventory Number, the date it is opened and an expiration date as appropriate. A Certificate of Analysis is obtained for reagents when available and archived in the QA Office.

Solutions prepared from reagents are recorded in the Reagent Preparation Logbook. The logbook includes a unique Reagent Number that is traceable to the Chemical Receiving Logbook. Reagent containers are labeled with Reagent Number, date of preparation, expiration date, and preparer's identification.

Procedures for Reagent Receiving and Preparation are detailed in SOP 1013S.

#### **Trace Metals Acids**

To ensure the quality of acids, nitric and hydrochloric, used for trace metals analyses, only the highest quality, certified "metals free" acids are purchased. Each lot received is analyzed for purity prior to use in the laboratory to assure that it is acceptable for use. Whenever possible,





entire lots will be reserved for use exclusively by ARI. This minimizes the possibility of receiving contaminated or unacceptable acid.

### **Solvents**

To ensure the quality of solvents used for sample preparation and analysis, the highest purity of solvents required for sample processing will be used. Purity checks will be performed on solvent lots received by the laboratory. Only those solvent lots determined acceptable will be used for sample processing. Whenever possible, entire solvent lots will be reserved for use. This minimizes the possibility of receiving contaminated or unacceptable solvents.

### **Compressed Gases**

To reduce the possibility of system contamination, compressed gases and liquids used for operating analytical instrumentation will be of a specified purity level. Any cylinder suspected of introducing contamination into a system will be promptly replaced.

### **4.5 Computer Systems**

ARI maintains several data systems. These are used to automate such diverse functions as accounting, payroll, sales and marketing, sample receiving, instrument data collection, production of hardcopy and electronic data deliverables, intra- and internet applications and project management. Specific information about these systems is contained in Appendix C and various SOPs.

ARI maintains a Laboratory Information Management System (LIMS) that stores analytical data, calculates final results and produces final reports (both hardcopy and electronic). The LIMS system is the major data system used at ARI. A separate Software Quality Assurance Plan outlines the QA/QC procedures for the LIMS system.



## **SECTION 5: LABORATORY DOCUMENTATION AND RECORDS**

All laboratory operations and procedures performed during sample processing are documented in logbooks, notebooks and on laboratory forms and bench sheets. Analytical data and copies of paper documents are also stored electronically. Consistent use of standard documents throughout the laboratory ensures that all activities will be traceable and serves as objective evidence of the work performed.

All procedures performed at ARI will be detailed in Standard Operating Procedures (SOPs). Sample preparation and analysis SOPs will reference approved analytical methods and detail the actual procedures followed by ARI staff. SOPs for non-analytical activities will detail the procedures developed specifically for use at ARI.

### **5.1 Responsibilities**

All staff members are responsible for complete and accurate documentation of laboratory activities. Each laboratory section develops a comprehensive set of documents (bench sheets, forms, etc.) to record all activities performed in that section. All staff members are responsible for reviewing and understanding SOPs, and must sign a record to document this fact. The QAPM is responsible for maintaining control of laboratory documents and ensuring their consistent use.

To ensure that all documents, SOPs in particular, accurately reflect the activities performed at ARI, section supervisors and managers are required to review all documents annually and recommend changes to the QAP. The QAPM is responsible for coordinating document revisions and ensuring that all staff members have access to the most current laboratory documents.

### **5.2 Document Control**

ARI's Quality Assurance Program requires that all forms and SOPs used within the laboratory be monitored to ensure that only the currently approved version of the documents are in use, centrally organized, and readily available to all staff members. All documents will include a



revision date. The LQAP and SOPs will also have an effective date. The time between the revision and effective dates will be used for training and orderly implementation of changes.

Electronic copies of laboratory documents will be maintained as part of the quality assurance files. Each laboratory section maintains working copies of pertinent forms and SOPs. The QAPM coordinates the generation of new forms or SOPs and modifications to existing documents. Log number assignments will be as follows:

Laboratory Section	Form Number	SOP Number
Client Services	0001 - 0999	001 - 099
Computer Systems	1000 - 1999	100 - 199
Data Services	2000 - 2999	200 - 299
Extractions	3000 - 3999	300 - 399
GC Laboratory	4000 - 4999	400 - 499
Metals Laboratory	5000 - 5999	500 - 599
Conventional Laboratory	6000 - 6999	600 - 699
Volatile Organic Laboratory	8000 - 8999	700 - 799
Semi-volatile Laboratory	7000 - 7999	800 - 899
Quality Assurance Monitoring	10000 - 10999	1000 - 1099
GeoTech Laboratory	11000 - 11999	1100 - 1199

Document numbers will include an F for forms and an S for SOPs i.e. 101F or 1234S. Document Control Logs of all forms and SOPs, detailing the form name and number, revision number and revision date will be maintained by the QA Officer. Outdated documents will be maintained in an electronic archive file.

The QAPM will distribute new and revised documents to the appropriate laboratory sections. Section staff will replace outdated copies of the document with the revised version. Laboratory forms and SOPs will be generated or revised on an “as needed” basis, and will be reviewed and revised as at least annually. Only the latest version of a form or SOP will be available in each laboratory. Section supervisors will periodically review these documents and recommend



changes to be implemented by the QAPM. A comprehensive review of all laboratory documentation will be performed annually at the direction of the QAPM.

To maintain document security, release of documents to clients or other outside agencies will be controlled by the QAPM. The QAPM will record the document to be released, revision number, person and agency receiving the document, and the release date. All documents generated by the laboratory will be considered proprietary. ARI permission must be obtained by anyone releasing the document to other agencies or including the document in a project or quality assurance plan.

### **5.3 Reference Documentation**

To provide an understanding of the procedures employed to generate quality data, a comprehensive set of reference materials is available to staff members. All activities performed within the laboratory can be referenced to a method or SOP. The laboratory maintains copies of the following method compilations:

*Code of Federal Regulations (Section 40)*

*Test Methods for Evaluating Solid Waste (USEPA SW-846)*

*USEPA Contract Laboratory Program Statement of Work for Organics Analysis*

*USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis*

*Methods for Chemical Analysis of Water and Waste (USEPA 500 and 600 series methods)*

*Standard Methods for the Examination of Water and Wastewater*

*Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound (PSEP)*

*US Naval Facilities Engineering Support Activity –NFESC (formerly NEESA).*

*Hazardous Waste Remedial Actions Program (HAZWRAP)*

*State of Alaska Department of Environmental Conservation (ADEC)*

*Oregon Department of Environmental Quality (DEQ) Petroleum Hydrocarbon Methods*

*Washington Department of Ecology (WDOE) Guidance for Remediation of Releases from Underground Storage Tanks (Appendix L)*

*Washington State SARA*

*AFCEE Project Quality Assurance Plan*

*Washington State EPH/VPH Methods*

*National Environmental Laboratory Accreditation Conference*

*Department of Defense Quality Systems Manual*

*Washington State Sediment Sampling and Analysis Plan*

Other methods followed within the laboratory are also available. Published modifications to analytical methods will be reviewed and incorporated into laboratory SOPs. If a method for a parameter is developed by ARI, it will be detailed in an SOP. SOPs will be available for all



laboratory activities. Each laboratory section will maintain a file or notebook of SOPs pertinent to that section. A compilation of all laboratory SOPs is maintained as part of the Quality Assurance Program files. A listing of laboratory SOPs is included as Appendix E.

The Quality Assurance Manual provides an overview of the laboratory-wide Quality Assurance program. A copy of the Quality Assurance Manual is distributed to all laboratory sections. Distribution of the QAP is coordinated by the QAPM.

ARI maintains a file of various laboratory and environmental publications and reference texts. These reference materials are available to all staff members. Operation and maintenance manuals are available for all equipment and instrumentation used within the laboratory. Additionally, senior level staff members are available to serve as reference sources. These staff members have numerous years of pertinent experience and can provide insight and guidance for all procedures and laboratory activities.

#### **5.4 Quality Assurance Policies**

Quality Assurance Policies provide standards and procedures to guide ARI employees in proper implementation of the QA Program. Appendix P includes current QA Policies.

#### **5.6 Worksheets and Logbooks**

##### Use of Laboratory Forms and Logbooks

All activities noted on laboratory forms and logs will be recorded in blue ink. Initials of the staff member performing the activity, as well as the date the activity is performed will be noted on all forms and logs. Any supplementary information about the activity, such as unusual observations or suspected procedural errors will be noted on the forms and logs. Laboratory logbooks will be prepared and controlled by the QAPM or his/her designee.

Changes to existing information will be annotated by drawing a single line through the original entry and initialing and dating the deletion. Correct information will be written above the deleted entry. When appropriate to clarify the intent of the change a note describing the reason for the change will be added. The use of correction fluids or other techniques that cover the entry in its entirety are not used on laboratory documents.



Since sample processing within an analytical laboratory involves many detailed steps, documentation can be quite extensive and varied. The following guidelines will be followed to encourage consistency in laboratory record keeping:

#### Standard Logbooks

Preparation of all stock and working standards is documented in the appropriate standards logbook. Each entry includes preparation date, initial and final concentrations (including solute and solvent amounts), standard ID number, expiration date and the identity of the person preparing the standard. Stock solution entries include standard lot number and supplier. Working solution entries include the stock solution ID number. Commercially prepared stock standards are recorded in the stock standard logbook.

#### Sample Storage Temperature Logs

The temperature of all refrigerators and freezers used for sample and standards storage is monitored daily. The temperature and recorder's initials are recorded on the temperature log attached to each unit. The acceptable temperature range for each unit is noted on the log sheet. Any out of control temperatures and/or corrective actions, must be noted on the log sheet and reported to appropriate personnel (Lab Supervisor and QA Manager)

#### Balance Calibration Logs

The true and measured values for each calibration check weight are recorded, along with the date and recorder's initials. Any actions taken, such as notifying the QAPM of malfunctions is indicated alongside the entry for that date.

#### Instrument Logs

The Instrument Run Logs must detail all samples analyzed on a given instrument for a given parameter. Instrument conditions, analysis date, analyst initials and standard or sample identifications in the analytical sequence must be recorded in the log. Comments related to sample analysis and minor maintenance are noted on the instrument logs. For GC/MS analyses, instrument performance is documented by recording internal standard response alongside the sample identification.



### Sample Preparation/Analysis Worksheets

Sample preparation and analysis activities are documented on appropriate worksheets. Sample identifications, weights or volumes used, intermediate cleanups, final volumes, preparation dates and analyst initials will be noted as well as any observations about sample condition. Any issues encountered during sample preparation are also noted. Surrogate and spiking solution ID numbers, and concentrations added to the samples, must be indicated on the bench sheet.

For some parameters, analytical results are summarized on an analysis worksheet. Sample identifications, sample preparation information, sample results, quality control results, analysis date, analyst initials and reported detection limits must be indicated on the worksheet. Any necessary data qualifiers are also noted on the worksheet.

### Maintenance Logs

All major maintenance performed on instrumentation or laboratory equipment must be documented. Maintenance performed, date and analyst performing the maintenance, and steps taken to verify that the maintenance was successful are detailed in the log. Routine maintenance of GC-MS instruments is documented on “maintenance cards” attached to each instrument. The demonstration that GC instruments are in-control following maintenance is documented in the instrument run log.

### Individual Laboratory Notebooks

Staff members preparing USEPA CLP samples must maintain unique laboratory notebooks for these analyses. Each case submitted is documented on a separate, sequentially numbered page. A listing of all samples prepared as part of the case, the date and the preparer’s initials, and any notes specific to sample preparation must be annotated in the logbook. Individual notebooks are used only when required by a specific contract. All sample preparation information is recorded on a laboratory bench sheet.



## **5.5 Document /Data Storage and Archival**

### Logbooks

All active logbooks will remain in the appropriate laboratory sections. Completed logbooks will be forwarded to the QAPM for archival.

### Magnetic Tapes and Diskettes

When instrument capabilities permit, all data generated is archived and stored on magnetic tapes or disks. The electronic media remains on file for approximately two years.

### Chromatograms and Instrument Documentation

Electronic or paper copies of chromatograms, instrument calibrations, quantification reports and any other printed documentation generated during sample analysis are maintained as part of the permanent data files. All hardcopy data remain on file at ARI for five (5) years or longer as specified by contract.

### Project Data and Documentation

Project data and support documentation, electronic or paper copies, will be filed a minimum of five (5) years, or as specified by contract.





## **SECTION 6: SAMPLE CONTROL**

All samples analyzed by the laboratory will be monitored in accordance with sample control procedures. Sample control includes operations such as container preparation, sample collection, receipt and storage, and tracking of the sample throughout all processing steps. Documentation of all sample control activities and adherence to standard procedures is an important aspect of ensuring that data quality objectives are met.

### **6.1 Sample Collection**

Production of quality analytical data begins with proper sample collection. Improper sampling procedures may result in inaccurate final results. Although the laboratory is not routinely involved with sample collection, it will minimize the possibility for error by providing clients with appropriate sample containers and sampling instructions for the requested parameters. If, upon receipt, sample integrity appears to be compromised, the client will be immediately notified to allow for re-sampling if necessary.

### **6.2 Sample Container Preparation and Shipment**

To minimize the possibility of contamination from containers furnished by outside sources, the laboratory will furnish all necessary sample containers for client projects when requested by the client. Sample containers, pre-cleaned to EPA specifications, or certified clean by the manufacturer or ARI, are supplied for most parameters. Containers for special purposes may be acquired upon request. Lot numbers for containers are tracked to link bottle orders to lot numbers.

A blank sample label is affixed to each sample container prior sending the container to a client. The sample label allows for recording of the following information at the time of collection: client name, client sample identification, sampling site, date and time of sample collection, analytical parameters, and any preservatives used. Sample labels provided by ARI are coated to prevent bleeding of recorded information if labels become wet.

To ensure that the correct number of appropriate sample containers are prepared and submitted to the client, a Bottle Request is completed by a Client Services staff member or Project Manager at the time sample containers are ordered by the client. All necessary



preservatives are also noted on the Bottle Request. The Bottle Request is then forwarded to appropriate personnel in the Sample Receiving Section for order preparation. All required containers will be gathered and preservatives added as specified. A copy of the Bottle Request accompanies the sample containers to allow the client to verify that the order is properly filled. Additional containers will be supplied for quality control purposes and in case of container breakage or sampling complications. A complete listing of containers and preservatives used within the laboratory is included as Appendix F.

To facilitate transportation of containers to the sampling site, sample containers will be placed in coolers along with appropriate packing material. The inclusion of packing materials, such as vermiculite or “bubblewrap”, is provided to minimize the possibility of container breakage and cross-contamination. Sample containers will be organized in the coolers per analytical or client specifications. Depending on client preference and project requirements, coolers and sample containers will be shipped to a specified location, delivered by ARI courier, or held at the laboratory for pick up. To ensure that sample identification, analytical parameters, and sample custody are properly documented, Chain of Custody records will accompany all sample container shipments. When appropriate, as for drinking water source sampling events or for parameters that require preservation in the field, sample collection instructions will also be included with shipments.

### **6.3 Sample Admission**

All samples received by the laboratory are processed in a central Sample Receiving area. To ensure the safety of staff members receiving samples, coolers will be opened under a hood or in a well-ventilated area. Appropriate protection, such as disposable gloves, safety glasses and laboratory coats will be worn during sample receipt and log-in. Additionally, all general safety practices as specified in ARI’s Chemical Hygiene Plan will be employed.

Upon receipt, sample coolers will be inspected for general condition and custody seals. Time and date of sample receipt, as well as identification of the staff member receiving the samples, will be indicated on each Chain of Custody record accompanying the shipment. Cooler temperatures will be determined using an IR temperature measuring device or by placing a thermometer in the cooler immediately after the cooler is opened. If samples cannot be logged-in within 30 minutes after receipt, the sample coolers will be tagged and placed in the



walk-in sample storage refrigerator for short-term storage. Chain of Custody records for the stored coolers will remain in Log-In to ensure that processing of the stored samples is not overlooked.

Samples to be processed will be removed from the coolers and organized by sample identification. The number and type of sample containers received will be verified against the Chain of Custody record. Each sample container will be examined to verify that the condition is acceptable and that sample integrity has not been compromised during shipment. Sample containers broken during shipment should be handled according to procedures detailed in the Chemical Hygiene Plan (Section 5, Waste Disposal Procedures).

After sample organization and initial inspection has been completed, sample information will be entered into the LIMS, and a Service Request will be generated for the sample set. The Service Request serves as a work order for the laboratory. The Service Request will contain the following information:

*Client Name*  
*Client Project Name and/or Number*  
*Client Contact*  
*Verified Time of Sample Receipt (VTSR)*  
*Required Turnaround Time*  
*Laboratory Job Number*  
*Client Sample Identifiers(s)*  
*Laboratory Sample Number(s)*  
*Required Parameters*  
*Additional Analytical Requirements/Comments*

Also entered into the LIMS are the number of sample containers for each sample, sample conditions, and cooler temperatures.

A sequential laboratory job number will be assigned to each sample set. Laboratory sample numbers, determined by the job number and a sequential letter, will be assigned to each sample. Containers for each sample will also be numbered sequentially. The accuracy of sample container labeling is verified by a second person. These identifiers will be used to monitor the sample set and container throughout sample processing. All samples logged for the sample set and the analytical parameters required for each sample will be indicated on the Service Request. Client specific quality control requirements and any other pertinent



information indicated on the Chain of Custody Record will also be noted. Discrepancies between the Chain of Custody record and sample containers will be noted, as well as discrepancy resolutions. To reduce the possibility of inaccurate sample processing, the sample receiving staff working with the Project Manager will resolve all noted discrepancies prior to releasing the samples to the analytical sections.

Upon completion of sample log-in, all documentation will be placed in a master folder and forwarded to the assigned Project Manager for review and approval. The master folder will be color-coded as follows:

Master File Color	Designation
Red	Accelerated Turnaround ( $\leq$ week)
Clear	Routine Turnaround

The Project Manager will review all aspects of the documentation, specify any additional analytical requirements and resolve any remaining discrepancies before sample processing begins. After Project Manager final approval has been obtained (indicated by the Project Managers initials and the date on the Service Request and laboratory-specific parameter sheets), the master file will be returned to Log-In for preparation of laboratory job folders. A job folder will be created for each laboratory section involved in sample processing for a given project. Laboratory job folders are color-coded as follows:

Job Folder Color	Designation
Red	Accelerated Turnaround (< 7 days)
Yellow	Accelerated Turnaround (7-14 days)
Orange	Organic Extractions (Routine TAT)
Blue	NWTPH-HCID Analyses
Neutral (manila)	>14 day TAT
Pink (or other)	Client Specific



Copies of the Service Request and all pertinent laboratory-specific documentation required to accurately complete sample analysis will be placed in each laboratory job folder. Laboratory job folders will then be distributed to appropriate laboratory sections for analysis and incorporation into the section tracking system.

### Subcontracting Policies

In some instances, ARI cannot perform certain analyses due to current laboratory workload limitations or specific analytical equipment requirements. In these instances it becomes necessary to subcontract work to other laboratories. In order to guarantee that data quality and defensibility are of the same high standards that ARI strives to achieve within our laboratory, policies regarding selection and use of subcontractor laboratories have been established as follows:

1. ARI's client must be made aware that samples will be subcontracted and what laboratory they will perform the analyses.
2. The sample information and analytical requirements are first entered into the ARI LIMS in the same way that samples for in-house analyses are processed. Subcontractor laboratories are contacted to verify their preparedness, and samples are then submitted to them using ARI chain-of-custody forms. These chain-of-custody documents are included in the master folder for the project.
3. Subcontractor laboratories must qualify to perform the analyses using the same criteria applied to ARI. When appropriate, subcontracted laboratories may be asked to submit documentation such as proof of certification or accreditation, quality assurance plans, standard operating procedures, results of method detection limit studies, control limits to ARI. ARI may at its discretion perform an on-site assessment of subcontracted laboratories. Failure to submit requested documents or refusal of an on-site assessment will disqualify laboratories from subcontracting ARI sample analyses.
4. ARI may request that subcontract laboratories analyze, on double blind performance testing (PT) sample obtained from commercial vendors at the subcontractor's expense.



5. The laboratory must be willing to maintain an annual contract with ARI, and must list ARI as a co-insured on the subcontract laboratory's liability insurance policies. Financial stability is also evaluated on a lab-by-lab basis.

#### **6.4 Sample Custody**

To ensure the traceability of sample possession, chain of custody is documented from sample collection to completion of final analysis, and is maintained during sample storage in archive prior to disposal. This is achieved through completion of a written chain of custody record. Custody of all samples and extracts processed by the laboratory is documented at each step of the analytical process.

The National Enforcement Investigations Center (NEIC) of EPA defines custody in the following ways:

*It is in your actual possession, or  
It is in your view, after being in your physical possession, or  
It was in your possession, then you locked or sealed it up to prevent tampering, or  
It is in a secure area.*

Sample handling may vary and specific custody procedures have been developed for each laboratory section.

#### Custody at Sample Log-in

A Chain of Custody Record must accompany all samples received by the laboratory. This record documents all sampling activities as well as persons handling the samples prior to receipt by the laboratory. Sample receiving staff assumes custody of samples upon receipt from the client or courier. Samples will remain in the custody of Sample receiving until the samples are delivered to a laboratory section. Should samples require shipment to a subcontracting laboratory, a separate Chain of Custody Record will be completed to document the sample transfer. Chain of Custody records will be included with sample data reports in the final analytical package submitted to the client. Copies of these records will be filed with project data.



### Custody of Volatile Organic Analysis (VOA) Samples

Upon completion of sample the sample receiving process, samples requiring analysis for volatile organic analysis will be placed in the VOA refrigerator (freezer) designated for incoming samples and logged into the VOA sample receipt logbook. The samples are now in the custody of the VOA laboratory. To avoid possible cross-contamination of low level samples, those samples known or suspected to contain high levels of contaminants, will be stored in a separate refrigerator prior to analysis.

VOA Laboratory analysts complete the receiving process and move the samples to a refrigerator designated for “active” samples. Samples removed from storage for analysis are considered to be in the custody of the analyst responsible for sample processing. All samples to be analyzed will be listed in the analytical logbook for the selected instrument. Laboratory and client sample identifications, the bottle number and Identification of the analyst performing the analysis will be indicated in the logbook. If it is necessary for sample custody to be transferred to another instrument or analyst, the second analyst will record this information. Thus, custody of a given sample can be traced throughout the analytical process, regardless of the number of instruments or analysts involved. Analysts will initial all raw data generated from sample analysis, to further document sample custody.

After completion of sample analysis, soil and intact water sample containers will be placed in the refrigerator designated for sample archival. Any water sample remaining in the container after completion of analysis will be considered compromised and will be discarded. The samples will remain in archive and in the custody of the VOA laboratory until final disposal.

### Custody of Semi-volatile Organic Analysis (SVOA) Samples

Upon completion of sample log-in, samples requiring extraction for organic parameters will be placed in walk-in cooler number 5. All samples placed in the cooler will be logged into the *Walk-in Admission Logbook*. Removal of samples from the refrigerator for processing by Extractions or Conventional personnel must be indicated in the *Walk-in Admission Logbook*. Samples stored in this walk-in refrigerator remain in Log-In custody until removed to a laboratory for processing.



The analyst responsible for the custody and initial handling of samples within the sample preparation laboratory will be indicated on the Sample Preparation Worksheet. All analysts involved in the subsequent steps of sample processing will also be indicated on the worksheet. Residual sample volumes will be archived in the refrigerator designated for extractable organic samples. Transfer of residual samples to this refrigerator will be documented in the *Sample Archive Refrigerator Logbook*. Transfer of prepared sample extracts to the appropriate analytical sections will be documented in the Extract Log in the preparation laboratory and in the Extract Log in the analytical section. Upon extract transfer, the analytical section receiving the extract assumes custody.

Extracts removed from storage for analysis are considered to be in the custody of the analyst responsible for analysis. Removal of extracts for analysis will be indicated in the Extract Log in the analytical section. All extracts to be analyzed will be indicated in the analytical logbook for the selected instrument. Laboratory and client sample identifications, as well as the analyst performing the analysis will be indicated in the logbook. Analysts will initial raw data generated from extract analysis to further document sample custody. After completion of analysis, extracts will be placed in the refrigerator designated for archive. Extracts will remain in storage and in the custody of the analytical section until final disposal.

#### Custody of Inorganic and Metals Samples

Upon completion of the sample receiving process, samples requiring preparation or analysis for inorganic parameters will be placed in the designated walk-in cooler. Selected samples such as those requiring a critical analysis are placed directly in the laboratory. Removal of samples from the refrigerators for digestion and/or analysis will be indicated in the *Walk-in Admission Logbook* for the appropriate refrigerator. Samples stored in the walk-in refrigerators remain in Log-In custody until the laboratory removes the samples for processing.

The analyst responsible for custody and initial handling of samples within the metals preparation laboratory will be indicated on the Sample Digestion Worksheet. All analysts involved in the subsequent steps of sample processing will also be indicated on the worksheet. Transfer of completed sample digests to the metals instrument (analysis) laboratory will be





documented by the metals preparation laboratory. Upon transfer of digests, custody is considered to be the responsibility of the analytical section receiving the digests.

Digests removed from storage are considered to be in the custody of the responsible analyst. All digests to be analyzed will be indicated in the analytical logbook for the selected instrument. Laboratory sample identifications and the analyst performing the analysis will be indicated in the logbook. If it is necessary for digest custody to be transferred to another instrument or analyst, the second analyst records this information. Thus, custody of a given digest can be traced throughout the analytical process, regardless of the number of instruments or analysts involved. Analysts will initial all raw data generated from digest and analysis to further document sample custody. After completion of analysis, digests will be stored by and remain in the custody of the analytical laboratory personnel until final disposal.

The analyst performing the sample analysis will remove samples requiring analysis for other inorganic (conventional) parameters from storage. Removal will be documented in the *Walk-in Admission Logbook*. Custody of the sample will be considered to be the responsibility of that analyst. All samples to be analyzed will be indicated on the worksheet for the required parameter. Laboratory sample identifications and the analyst performing the analysis will be indicated on the worksheet. If it is necessary for sample custody to be transferred to another instrument or analyst, the second analyst will record this information. Thus, custody of a given sample can be traced throughout the analytical process, regardless of the number of instruments or analysts involved. The analysts' initials will be indicated on the worksheet to further document sample custody.

### Special Chain of Custody Requirements

Should a client project require additional or more detailed custody documentation, requirements will be incorporated into the procedures for that project. Samples processed as part of the USEPA Contract Laboratory Program require more stringent chain of custody procedures. For this program, removal of samples and extracts for analysis (or any reason) will be documented in the Sample Control Log. Date, time and reason for removal, and date and time of return, will be fully documented. Removal of samples or extracts for permanent archiving or disposal will also be fully documented in the Sample Control Log.



## 6.5 Sample Archival and Disposal

After completion of analysis, unused sample aliquots are routinely stored for a specified period of time: 30 days for water samples and 60 days for soil samples. Colored markers are placed on samples with specific storage requirements during the sample receiving process. The color-coding is defined in the following table:

Label Color	Storage Requirement
Red	Hold until further notice
Orange	Suspected Hazardous
Yellow	Shared Sample containers
Blue	Samples to be frozen

Samples submitted for archival will be logged into the Sample Archive Logbook. Laboratory and client identifications, as well as archive date will be indicated in the logbook. The anticipated disposal date for the sample set will also be noted. The logbook will be reviewed several times during each week to determine samples scheduled for disposal. On or soon after the scheduled disposal date, the samples will be removed from archive storage and disposed.

In consideration of disposal requirements for hazardous samples, each sample processed by the laboratory will be evaluated for contamination levels based on final analytical results. Those samples containing analytes of interest at or above regulated disposal levels will be identified and handled as hazardous waste. A designated staff member coordinates periodic pickup and disposal of hazardous waste by an USEPA approved TSD (Treatment, Storage, and Disposal) Company and maintains hazardous waste disposal records. Specific guidelines for handling hazardous samples and waste are detailed in the Chemical Hygiene Plan (Section 5, Waste Disposal Procedures)

## **SECTION 7: PROJECT MANAGEMENT AND TRACKING**

### **7.1 Project Management**

Concise and accurate communication between a client and ARI, and within the laboratory, is an extremely important requirement for generating quality analytical results. All clients contracting with ARI will be assigned to a Project Manager. The Project Manager confirms that project requirements are consistent with laboratory capabilities, and coordinates with laboratory sections to provide analytical results within specified project timelines. Project organization, monitoring, and follow-up is the responsibility of Project Management staff.

Client project requirements and Project Managers' areas of expertise will be considered for client assignment. To ensure that all clients and projects receive the attention necessary for successful project completion, Project Manager workloads will also be considered. Project Managers will serve as the central focus for all project related activities and communications.

The Project Manager will review work plans and requirements for all pending projects. Any questions related to the work plan will be addressed prior to project commencement. The Project Manager will consult with appropriate analytical sections to clarify any issues regarding procedures and capabilities. Project deliverables requirements will also be addressed at this time. Upon receipt and log-in of project samples, the Project Manager will review all documentation to ensure that samples were properly logged in, and that analytical and QC requirements were correctly specified. The Project Manager will also provide any additional project related information that will assist the analytical sections with sample analysis. Laboratory sections will not process a sample until Project Manager approval has been given. Exceptions are parameters with critical (less than 48 hour) holding times or those that arrive on weekends or holidays when none of the Project Managers can be contacted.

Throughout the project, the Project Manager will monitor all analytical activities to help ensure that the project is completed and delivered on schedule. Any issues arising during sample processing will be promptly discussed with the client. Likewise, the analytical staff will be informed of any client concerns or project modifications. The Project Manager will also address any issues that arise during subsequent review of the analytical data by the client.



## **7.2 Project Tracking**

Monitoring the laboratory workload ensures that adequate staffing and equipment will be available to produce quality analytical data and meet client needs. At the time a client project is tentatively scheduled, information regarding the project will be documented in the Project Management Database. Project particulars, sample quantities, parameters and anticipated sample delivery dates will be specified, as well as any prearranged analytical costs. Project work plans and any other project information will be kept on file with the Project Manager. Schedules for pending projects are communicated to the lab sections through periodic distribution of database printouts. Upon receipt of project samples, the project Inquiry number will be referenced to ensure project requirements are accurately specified. The original project documentation will be placed in the master folder as part of the project file.

Each laboratory section analyzing project samples will be responsible for ensuring that all analyses are accurately completed by the required date. All staff members are required to be aware of holding times, special analytical requirements, and required turnaround times. Analytical sections will remain in close communication with the Project Management staff so that any issues arising during sample analysis can be promptly addressed or discussed with the client.

Project Managers or their designee are responsible for monitoring project status. Sample status reports are generated as needed from LIMS and are distributed to lab sections and Project Managers. These reports allow the Project Managers to review project status and identify any samples which must be expedited to meet project timelines. Additionally, verbal communication between Project Managers and lab sections provides information about project status.

After sample analysis, report generation, and final review have been completed, data and final reports will be forwarded to the Project Manager. If requested, preliminary and interim results will be forwarded to the client. When all final data are available, the Project Manager will assemble the final package, verifying that all analyses were completed and project requirements met. A project narrative detailing the particulars of sample processing will be generated. After assembly and prior to shipment, the Project Manager will perform a final, cursory review of the package for any inconsistencies or incorrect information. The package



will then be forwarded to clerical personnel for photocopying and shipment. The Project Manager will determine final analytical costs and submit this information to the Accounting department for invoicing. Upon completion, all raw data and documentation associated with each client project will be compiled and stored as part of the laboratory project files. A chart detailing laboratory workflow as described in this section is included as Appendix G.



## SECTION 8: ANALYTICAL METHODS

To ensure that all data generated are consistent and comparable, clearly defined procedures will be followed for all aspects of sample processing, control and management. Standard Operating Procedures (SOPs) provide detailed guidelines for completing a procedure. Document control procedures and periodic audits will ensure that operations are performed in accordance with the most current SOPs. All routine deviations from published will be noted in the SOPs. Analysis specific deviation will be noted in Analyst Notes and in the Analytical Narrative.

### 8.1 Responsibilities

It is the responsibility of staff members to perform all procedures in accordance with the guidelines specified in the Standard Operating Procedures. Laboratory management is responsible for ensuring that SOPs are followed throughout the laboratory. The QAPM is responsible for coordinating periodic review and revision of existing SOPs and generation of additional SOPs. The QAPM is also responsible for maintaining SOP document control and ensuring that the most current versions of all SOPs are available to staff members.

### 8.2 Methods

Laboratory procedures may reference any established methods specified in the following publications:

1. *Code of Federal Regulations (Section 40)*
2. *Test Methods for Evaluating Solid Waste (USEPA SW-846)*
3. *USEPA Contract Laboratory Program Statement of Work for Organic Analysis*
4. *USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis*
5. *Methods for Chemical Analysis of Water and Waste (USEPA 500 and 600 series)*
6. *Standard Methods for the Examination of Water and Wastewater*
7. *Protocols for Measuring Selected Environmental Variables in Puget Sound (PSEP)*
8. *Navy Installation Restoration Laboratory Quality Assurance Guide (February 1996)*
9. *Hazardous Waste Remedial Actions Program (HAZWRAP)*
10. *State of Alaska Department of Environmental Conservation (ADEC)*
11. *Oregon Department of Environmental Quality (DEQ) Petroleum Hydrocarbon Methods*
12. *Washington Department of Ecology (WDOE) Guidance for Remediation of Releases from Underground Storage Tanks (Appendix L)*
13. *The Department of Defense Quality Systems Manual (DoD-QSM)*
14. *Washington State Sediment Sampling and Analysis Plan*



The laboratory will adhere to established methods whenever possible. Occasionally, however, procedures determined to provide more accurate final results will be incorporated into the method. Should the laboratory procedures deviate from the established method, all modifications will be detailed in the associated SOP. A listing of laboratory SOPs is included as Appendix E.

### **8.3 Standard Operating Procedures**

Standard Operating Procedures (SOPs) are detailed, step-by-step instructions for completing a laboratory operation. SOPs will address all procedures within the laboratory, from initial project identification to final data archival. SOPs will be generated for procedures developed within the laboratory and for those that follow established methods.

To ensure consistency in defining procedural guidelines, all SOPs will contain the following sections:

*Scope and Application*  
*Definitions*  
*Equipment*  
*Documentation and Forms*  
*In-house Modifications to Referenced Method*  
*Procedures*  
*Review*  
*Quality Control*  
*Corrective Actions*  
*Miscellaneous Notes and Precautions*  
*Method References*  
*Appendices*

SOPs will be monitored through the laboratory document control system. Each SOP will be assigned a document control number as detailed in Section 5.2 of this LQAP. SOPs are revised whenever a laboratory procedure is changed or modified. All SOPs are reviewed and revised as necessary at least once a year. Personnel normally performing the procedure or analysis perform the review. SOPs will be generated for each new procedure implemented within the laboratory. Review, modification, new SOP generation, and distribution will be coordinated through the QAPM. The QAPM will periodically audit the laboratory sections to verify that the most current versions of all SOPs are in use. Document release will be controlled as detailed in section 5.2.



#### **8.4 Method Selection and Use**

Method selection will be based on availability of analytical instruments and equipment, chemical standards, expected method performance and marketability. Methods that are defined and accepted by regulatory agencies and familiar to ARI's clients are preferred. The Laboratory Manager and QAPM in consultation with marketing, client service, and laboratory supervisory staff are responsible for selecting appropriate methods. Client or project-specific methods may be used when appropriate.

The most recently promulgated method will be used for all procedures. Non-promulgated methods will be investigated if requested by a client. Section supervisors and managers are responsible for ensuring that the procedures in use reflect the requirements of the promulgated methods. Any modifications made to the method must be documented in the SOPs. Method modifications may be acceptable, provided all acceptance criteria specified in the method are met.

Section supervisors and managers review newly promulgated methods. SOPs will be modified as necessary to reflect the new methods. When possible, the annual SOP review will be coordinated with anticipated method promulgation dates. This is especially useful for large method compilations, such as SW-846. If the annual SOP review and method promulgation cannot be coordinated, SOPs will be revised as soon as possible after a method has been promulgated, especially when method changes are significant.

SOPs will be generated to reflect the most commonly used methods and protocols. If more than one method is used for an analysis, separate SOPs should be generated. Several methods may be incorporated into one SOP, provided that each method is clearly identified and defined in the SOP. Method modifications or special requirements for ongoing projects, or for specific programs (Navy, CLP, etc.), will be incorporated into the SOP. These requirements will be annotated to indicate that they are project/program specific. Analysts and technicians will be responsible for ensuring that, when required, project or program specific procedures are followed. SOPs will be controlled as specified in section 5.2.





## **8.5 Method Performance**

Method performance must be demonstrated for all new methods prior to using methods for sample analysis. Section supervisors and managers are responsible for ensuring that method performance is demonstrated and support procedures have been performed.

Method performance will be demonstrated in the following manner:

- A draft SOP will be generated for the method. The SOP must provide sufficient detail to perform the analysis and must accurately reflect the published method. Any steps in the method for which analyst discretion is allowed must be clearly defined.
- A method detection limit (MDL) study must be performed for the method. Method detection limits must be verified to be at or lower than any method-specified detection limits. Method detection and reporting limits must be established.
- Method precision and accuracy must be evaluated. This may be determined using an MDL or IDL study. Replicates will be evaluated for precision; analyte values will be compared to spike amounts to determine accuracy. Any method-specified precision and accuracy criteria must be met.

All method performance results will be reviewed and compiled by the section supervisor. Results will be filed with the QA section. A final SOP will be generated and distributed.



## SECTION 9: INSTRUMENT CONTROL

### 9.1 Detection Limits

To verify that reported limits are within instrument and method capabilities, three levels of detection have been established: instrument detection limits, method detection limits, and reporting limits. Instrument and method detection limits are statistically based values, determined from replicate analyses of analytical standards. Reporting limits are based upon the experience and judgment of an analyst. Reported values will be qualified based on the established limits. All limits will be summarized and controlled by the QAPM and are included as Appendix I.

#### Instrument Detection Limits

The instrument detection limit (IDL) is considered to be the smallest signal above background noise that an instrument can reliably detect. This limit reflects whether or not the observed signal has been caused by a real signal or is only a random fluctuation of noise from the blank. The IDL does not take into consideration the performance or efficiency of analytical methods.

Instrument detection limits are determined annually, or when ever a major change has been made, for each instrument in the metals analysis laboratory. Seven replicates, of a blank, or standards containing analytes at levels three to five times the expected IDLs are analyzed on three non-consecutive days. The IDL value for an analyte is three times the average of the standard deviations from the three replicate sets of analyses.

#### Method Detection Limits

The method detection limit (MDL) is considered to be the lowest concentration of an analyte that a method can detect with 99% confidence. Method detection limits will be established for all analytical parameters according to the guidelines specified in the Code of Federal Regulations, Section 40. Seven replicate samples are fortified with target analytes at levels that are one to five times (but not exceeding 10 times) the expected detection limits. The MDL for an analyte is determined to be the standard deviation of the replicates times the appropriate student's t-test value. More than seven replicates may be processed, but all replicates must



be used in the MDL determination. To report data without qualification, statistically determined MDLs cannot exceed any method specified MDLs.

Laboratory supervisors or managers review all statistically determined MDLs for accuracy and validity. The section supervisor or manager is responsible for ensuring that any unusable MDL studies are reprocessed. Once accepted, MDL study results and associated raw data will be forwarded to the QA section for further review and additional approval. MDLs approved by both section management and QA will be considered final and acceptable for use. Finalized MDL values are forwarded to Computer Services for incorporation into ARI's LIMS.

MDL studies will be conducted for all analyses performed by the laboratory on representative water, sediment and, tissue samples when appropriate and suitable sample matrices are available. MDL studies for inorganic analyses are only performed on aqueous samples. MDL studies will be performed on all instruments used for sample analysis. To allow for reevaluation of method performance, MDL studies will be performed on an annual basis. The QAPM is responsible for ensuring that all MDL studies are performed at least annually. Section supervisors and managers are responsible for determining if and when additional MDL studies should be performed due to changes in analytical methods, instrumentation or personnel.

### **Reporting Limits**

Reporting Limits (RL) are the lowest quantitative value routinely reported. Analytical results below the RL will be expressed as "less than" the reporting limit. RLs are estimated values based upon the MDLs, experience and judgment of the analyst, method efficiency, and analyte sensitivity. No reporting limit will be lower than its corresponding MDL. RLs will be verified on a regular basis either by having a calibration standard at the limit or by analyzing a standard at the RL immediately following initial calibration.

### **Analytical Standards**

Generation of high quality results is dependent upon the use of accurately prepared analytical standards. Many stock standards used within the laboratory are commercially prepared solutions with certified analyte concentrations. Neat standards used for stock standard



preparation are of the highest purity obtainable. Standard preparations are fully documented in appropriate logbooks.

### Responsibilities

It is the responsibility of each laboratory employee involved with standards preparation to ensure that all standards are correctly and accurately prepared through the use of good laboratory practices and analytical verification. It is also the responsibility of these staff members to properly document the receipt and/or preparation of all standards. Management is responsible for ensuring that all staff members follow specified standards preparation and inventory procedures. The QAPM is responsible for periodically auditing standard preparation records to verify compliance with the laboratory Quality Assurance Program.

### Organic Standards Preparation

Two types of standards are utilized for extractable organic compounds: neat standards from which stock solutions are prepared, and commercially prepared stock solutions from which working solutions are prepared. The type of standard depends upon availability. Commercially prepared standards are preferred when available.

Preparation of stock solutions will be documented in the Stock Solutions Log. To ensure traceability, commercially prepared stock solutions will also be documented in the Stock Standard Solutions Log. Each solution will be assigned a unique stock number determined by the page number and entry number on the page, preceded by "S" to indicate the solution is a stock, volatile stock standard are labeled "VS". For example, the third entry on page 44 will be assigned the stock number S44-3. For stock solutions prepared from neat standards, the compound, supplier, lot number, preparation schematic, preparation date, expiration date, and analyst initials will be recorded. After preparing the standard, another analyst should review the preparation information to verify accuracy. For commercially prepared stock solutions, the compound, supplier, lot number and expiration date will be recorded. As a stock solution is not actually prepared in-house for these commercial solutions, it is not necessary to record or verify a preparation schematic.

Preparation of working solutions (including spike and surrogate solutions) will be documented in the Working Standard Solutions Logbook. Each solution will be assigned a working



standard number determined by the page number and entry number on the page. For example, the second entry on page 73 will be assigned the working standard number 73-2. For volatile organic standards, the working standard number is preceded by "VW". The compound, stock solution reference, preparation schematic, preparation date, expiration date, and analyst initials will be recorded. After preparing the standard, another analyst will review the preparation information to verify accuracy. After analyzing the standard and confirming that it is acceptable, analytical verification will be documented in the logbook.

Discarded or consumed standards will be annotated in the logbook by drawing a single line through the entry, indicating "discarded" or "consumed" above the line with confirming initial and date. Existing standard numbers will not be reused. Instead, each new stock or working solution made will be assigned a new number.

Standards preparation will be performed in accordance with good laboratory practices. Syringes, glassware and other preparation equipment will be thoroughly cleaned prior to and after use. Standard material weights and solution volumes will be accurate to  $\pm 3\%$ . Neat standards that are less than 97% pure must be corrected for concentration. Standard solutions will be stored in amber bottles with Teflon-lined caps. Each standard solution will be labeled with the solution number, compound, analyst initials and expiration date. Stock solutions will be stored in the appropriate standards freezer; working solutions will be stored in the appropriate standards refrigerator.

### Metals Standard Preparation

Commercially prepared single element stock solutions are used for all elements. Preparation of working solutions from these single element stocks will be documented in the Solutions Logbook. Preparation of check standards will also be documented in the Solutions Logbook. The element, preparation schematic, preparation date, expiration date, and analyst initials will be recorded. Working calibration standards are prepared weekly for furnace and ICP analyses and as needed for ICP-MS. Calibration verification standards are prepared daily for GFA analyses and as needed for ICP and ICP-MS analyses.

Standards preparation will be performed in accordance with good laboratory practices. All preparation equipment will be thoroughly cleaned prior to and after use.



### Inorganic (Wet Chemistry) Standard Preparation

Working standards for wet chemistry parameters will be prepared on a daily basis, prior to starting an analysis. Stock and check standard solutions will be replaced as solutions expire or are consumed. Stock and check standard solutions will be labeled with the compound, preparation data (weight and volume), units of concentration, preparation date, expiration date, and analyst initials.

Standards preparation will be performed in accordance with good laboratory practices. Glassware and other preparation equipment will be thoroughly cleaned prior to and after use. Standard material weights and solution volumes will be accurate to  $\pm 3\%$ . Stock standards will be stored in containers appropriate for the parameter.

### **9.3 Calibration**

Instrumentation and equipment used for sample processing and analysis must be operating optimally to ensure that accurate analytical results are generated. Verification of optimum operation is accomplished through various tuning and calibration procedures. Criteria for determining the accuracy of calibration are specified for all instrumentation and equipment. Prior to sample analysis, calibrations will be analyzed and evaluated against specified acceptance criteria. Acceptance criteria are either published as part of the method or generated at ARI using control charts. Calibration verifications will also be analyzed throughout an analytical sequence to ensure that instrument performance continues to meet acceptance criteria.

### **Gas Chromatography/Mass Spectrometry (GC/MS)**

All GC/MS systems will be evaluated through analysis of an instrument performance check solution and calibration standards. The composition of the standards varies depending on the analysis performed on the system. System evaluation will be performed prior to sample analysis. Evaluation criteria used for GC/MS analyses are as specified for the SW846 methods.

Instrument Performance Check Solution - Prior to analysis, the system will be evaluated to ensure that mass spectral ion abundance criteria are met. Bromofluorobenzene (BFB) is analyzed for volatile organic analyses and



Decafluorotriphenylphosphine (DFTPP) is analyzed for semi-volatile organic analyses. All ions must meet method-specified criteria.

The instrument performance check solution will be analyzed at a minimum of every 12 hours during the analytical sequence. Each analysis of the check solution will be verified against the specified criteria.

Calibration - After instrument performance has been verified, each GC/MS system will be calibrated to verify response linearity. For volatile organic analyses, up to eight standards ranging from 1 to 200 µg/L will be analyzed. For semi-volatile organic analyses, five to seven standards ranging from 2 to 80 µg/L will be analyzed. The standard levels evaluated will vary depending on the compound. Initial calibration results will meet percent relative standard deviation acceptance criteria.

A continuing calibration verification standard at a mid-level concentration (routinely 50 µg/L for VOA and 250 µg/L for SVOA) will be analyzed at a minimum of every 12 hours during the analytical sequence. For continuing calibrations, minimum response factor and percent difference criteria will be considered in evaluating the acceptability of the calibration. Initial and continuing calibration acceptance criteria for volatile and semi-volatile organic analyses are presented in Appendix J. All calibration data printouts will include the following documentation:

*Date of calibration,  
Identification of standard used  
Identification of person performing the calibration*

The analyst performing the calibration will include documentation of any problems encountered during the calibration analyses with the data, and will also note any corrective actions taken. The calibration data will be tabulated, and summary statistics will be generated. These results will be kept on file with the raw data in the Data Services section.

Internal Standard Responses - Internal standard responses and retention times in all standards will be evaluated immediately after analysis. This will serve as a baseline from which all sample internal standard responses and retention times will be evaluated.

### Gas Chromatography (GC)

Each GC and HPLC system will be calibrated to verify response linearity. Depending on the parameter, five to seven standards at concentrations covering the linear range of the instrument will be analyzed. Percent relative standard deviations for initial calibrations will not exceed SW-846 limits or 25% when those limits are not applicable.

A continuing calibration standard at mid-range concentration will be analyzed after every 10 samples or more frequently if the method or conditions warrant. Percent differences between



initial and continuing calibrations will not exceed SW-846 limits or 25% when those limits are not applicable.

Calibration for organochlorine pesticides will follow SW-846 guidelines. The initial calibration sequence specifies the analysis of Resolution Check, Performance Evaluation, five-point initial calibration, individual standards and instrument blanks. Criteria for evaluating these standards are as follows:

**Performance Evaluation** - The Performance Evaluation standard will be analyzed immediately following the Resolution Check standard. All standard peaks will be completely resolved. Individual breakdowns of DDT and Endrin will be less than or equal to 15% on both columns. A Performance Evaluation standard will also be analyzed at the end of the calibration sequence.

**Initial Calibration** - An initial calibration consisting of levels of standard concentrations will be analyzed immediately following the analysis of aroclor 1660 curve and individual aroclor and Toxaphene standards. The percent relative standard deviation (RSD) will not exceed SW-846 guidelines or 20% on each column.

**Continuing Calibration** - A midpoint Aroclor 1660 and or a midpoint pesticide standard along with a performance evaluation standard are analyzed after every ten (10) sample analyses. The continuing calibration standards will be within 85 - 115% of the initial calibration. The Performance Evaluation standard will meet previously specified criteria.

The analytical sequence may continue indefinitely, provided that calibration criteria are met throughout the sequence. Additionally, retention times for all compounds will fall within the retention time windows established by the initial calibration sequence of the three standard concentration levels.

All calibration data printouts will include the following documentation:

*Date of calibration,  
Identification of standard used, and  
Identification of person performing the calibration.*

The analyst performing the calibration will include documentation of any problems encountered during the calibration analyses with the data, and will note any corrective actions taken. The calibration data will be tabulated, and summary statistics will be generated.





## Metals

Analytical instrumentation for metals will be evaluated through the analysis of calibration standards, calibration blanks, and calibration verification standards. Initial calibrations will be performed prior to sample analysis.

### **Inductively Coupled Plasma Atomic Emission Spectrometry (ICP)**

Initial standardization is performed daily, or more frequently as required, by analyzing a blank and four multiple element standards with a single concentration for each analytical wavelength. The calibration is immediately verified with the analysis of an initial calibration verification standard (ICV) obtained from a source independent from the IC standard. The calibration will then be verified throughout the analytical sequence by analyzing a continuing calibration verification standard (CCV) after every 10 sample analyses. The calibration check standard values will be within  $\pm 10\%$  of the true value.

After initial calibration, a calibration blank (ICB) will be analyzed to check for baseline drift or carryover. The level of analyte in the calibration blank should be  $\pm 2$  RL. Calibration blanks (CCB) will be analyzed immediately following each calibration verification standard analysis.

Following calibration verification a standard at the reporting limit (CRI) is analyzed for all elements. Warning limits have been set at  $\pm 1$ RL and any sample determined to have a concentration below this standard will be reported as undetected.

The upper limit of the calibration range, linear dynamic range, is established for each analytical wavelength using standards of increasing concentrations. These standards are analyzed against the normal calibration curve and must be within 10% of their true value to verify linearity. At a minimum this upper range will be checked every six months or whenever major changes are made to the instrument. Any sample analyzed with a concentration above this linear dynamic range will be diluted and reanalyzed.

Also to verify the inter-element correction equations, inter-element correction standards (ICS) are analyzed both at the start and end of the analytic run. Both the major interfering and the interfered with elements are evaluated.

### **Atomic Absorption Spectroscopy (Graphite Furnace and Cold Vapor)**

Atomic absorption instrumentation is initially calibrated using a minimum of three standards of varying concentrations and a calibration blank. Initial calibration is performed daily or more frequently if conditions warrant. The calibration is immediately verified with the analysis of an independent source initial calibration verification standard (ICV). The calibration will then be verified throughout the analytical sequence by analyzing a continuing calibration verification standard (CCV) after every 10 sample analyses. The initial calibration verification standard



value will be within  $\pm 10\%$  of the true value whereas the CCV will be considered in control if it is within  $\pm 10\%$  for Graphite Furnace analysis or  $\pm 20\%$  for Cold Vapor analysis.

After initial calibration, a calibration blank (ICB) will be analyzed to check for baseline drift or carryover. The level of analyte detected in the calibration blank should be  $\pm 1$  RL. Calibration blanks (CCB) will be analyzed immediately following each calibration verification standard analysis.

Following calibration verification a standard at the reporting limit is analyzed for all elements. Warning limits have been set at  $\pm 1$ RL and any sample determined to have a concentration below this standard will be reported as undetected. Any sample determined to have a concentration above the high calibration standard will be diluted and reanalyzed.

### **Inductively Coupled Plasma Mass Spectrometry (ICP-MS)**

Initial standardization is performed daily, or more frequently as required, by analyzing a blank and four multiple element standards. The calibration is immediately verified with the analysis of an independent source initial calibration verification standard (ICV). The calibration will then be verified throughout the analytical sequence by analyzing a continuing calibration verification standard (CCV) after every 10 sample analyses. The calibration check standard values will be within  $\pm 10\%$  of the true value.

After initial calibration, a calibration blank (ICB) will be analyzed to check for baseline drift or carryover. The level of analyte in the calibration blank should be  $\pm 1$  RL. Calibration blanks (CCB) will be analyzed immediately following each calibration verification standard analysis.

Following calibration verification a standard at the reporting limit (CRI) is analyzed for all elements. Warning limits have been set at  $\pm 1$ RL and any sample determined to have a concentration below this standard will be reported as undetected.

The upper limit of the calibration range, linear dynamic range, is established for each analytical wavelength using high level standards. These standards are analyzed daily, or as necessary, against the normal calibration curve and must be within 10% of their true value to verify linearity. Any sample analyzed with a concentration above this linear dynamic range will be diluted and reanalyzed.

Also to verify the inter-element correction equations, inter-element correction standards (ICS) are analyzed both at the start and end of the analytic run. Both the major interfering and the interfered with elements are evaluated.

### **Inorganic Analyses other than Metals (Conventional Analyses)**

Instrumentation and equipment used in analyzing samples for conventional wet chemical parameters (predominantly inorganic anions and aggregate organic characteristics) will be evaluated through the analysis of either internally prepared primary standards or externally derived Standard Reference Materials.

Depending upon the analysis, calibration is based upon direct stoichiometric relationships, regression analysis, or a combination of the two. Stoichiometry generally involves standardization of a titrant against a known primary standard and then the use of that titrant for determining the concentration of an unknown analyte (e.g. the use of sodium thiosulfate in the iodometric titration of dissolved oxygen). Regression analysis involves the determination of the mathematical relationship between analyte concentration and the response produced by the measurement being employed. Regression analysis is used for colorimetric determinations, ion specific electrode analysis and ion chromatography. The curve of response versus concentration is fit by the method of least squares using linear, polynomial or logarithmic regression dependant upon the pattern of response being measured.

Calibration is repeated for each analytical batch. Immediately following calibration, the standardized titrant or the calibration curve will be verified by the analysis of an Initial Calibration Verification standard (ICV) and Initial Calibration Verification Blank (ICB). The verification standard will be derived from a source other than that used for standardization or development of the standard curve. The ICV must return a value within 10% of its known concentration. The ICB must be less than the Reporting Limit (RL) or the lowest point on the standard curve, whichever is less. Initial calibration verification must be successfully completed prior to the analysis of any samples.

Calibration verification will be repeated after every ten samples processed during an analytical run. This Continuing Calibration Verification (CCV) will validate the method performance through an analytical sequence. If the continuing calibration values for either the standard or blank are out-of-control, the analyst will verify the outlying condition and, if verified, the analysis will stop and the method will be re-calibrated. All samples run between the outlying CCV and the preceding in-control CCV will be re-analyzed. In-control verification standards and blanks must bracket all samples within an analytical run.

Initial calibration depending upon the analysis is based on either a direct stoichiometric relationship, a linear regression analysis or a combination of the two. Stoichiometry generally involves standardization of a titrant and use of that titrant for determining the concentration of an unknown analyte (e.g. the use of thiosulfate in iodometric determination of dissolved oxygen). Regression analysis involves the determination of the mathematical relationship between the analyte concentration and the response produced by the measurement being employed. The curve is fit by the method of least squares using a linear, polynomial or logarithmic regression depending on the response being measured. The regression coefficient will be greater than or equal to 0.995 for the calibration to be considered acceptable.

Initial calibration curve is verified throughout the analytical sequence by analyzing a calibration verification standard after every 10 sample analyses. The calibration verification standard value will be within  $\pm 10\%$  of the initial calibration.

After initial calibration, a calibration blank will be analyzed to determine target analyte concentration levels. The level of analyte detected in the calibration blank will be less than the lowest standard concentration in the initial calibration.



## **SECTION 10: DATA VALIDATION and REVIEW**

One hundred percent (100%) of laboratory data generated at ARI are subjected to a four level validation (review) process prior to release from the laboratory. The four levels of review are:

1. Analyst review
2. Peer review
3. Supervisory review
4. Administrative review

The data review process is outlined below and detailed in SOPs 200S through 206S.

In addition, Quality Assurance Personnel review 10% or more of all completed data packages for technical accuracy, project compliance and completeness. The data validation outlined below is completed in addition to the initial project review explained in Section 7 and QA specific reviews outlined in Section 11. If it is determined at any point during the analysis, reporting, or review process that data are unacceptable, prompt and appropriate corrective action must be taken. The corrective action will be determined by the situation. It is the responsibility of all staff members involved in data reporting and review to be aware of the quality control requirements and to be able to identify occurrences that require corrective action.

### **Analyst review:**

Each analyst is responsible for producing quality data that meets ARI's established requirements for precision and accuracy and is consistent with a client's expectation.

Prior to sample preparation or analysis an analyst will verify that:

1. Sample holding time has not expired.
2. The condition of the sample or extract is described accurately on the laboratory bench sheet.



3. Specified methods of analysis are appropriate and will meet project required Data Quality Objectives.
4. Equipment and Instrumentation are in proper operating condition.
5. Instrument calibration and/or calibration verification are in control.

During sample preparation or analysis an analyst will:

1. Verify that Method Blanks and Laboratory Control Samples are in control.
2. Verify that QC (replicate, matrix spike analyses, SRM, etc.) samples meet precision and accuracy requirements.
3. In addition to verifying that quality control requirements are met, the analyst will review each sample to determine if any compound of interest is present at levels above the calibrated range of the instrument.
5. Check for data translation or transcription errors
6. Record all details of the analysis in the appropriate bench sheet or logbook.
7. Note any unusual circumstances encountered.

Following the analysis or sample preparation an analyst will:

1. Examine each sample and blank to identify possible false positive or false negative results.
2. Determine whether any sample requires reanalysis due to unacceptable quality control.
3. Review data for any unusual observations that may compromise the quality of the data, such as matrix interference
4. Review and verify that data entry and calculations are accurate and no transcription errors have occurred.
5. Document anomalous results or other analytical concerns on the bench sheet, corrective action form or Analyst Notes for incorporation into the case narrative.
6. Note data with qualifying flags as necessary.



7. Enter reviewed data into LIMS as appropriate, incorporate all necessary sample and quality control information into the data package and forward it for further review.

**Peer review:**

A second analyst trained in the appropriate SOPs will complete a peer review. Peer review will include at a minimum:

1. Verification that all QA (holding times, calibrations, method blanks, LCS, spiked sample analyses, etc.) criteria are in control.
2. Examination the data for possible calculation and transcription errors.
3. Review bench sheets and analyst notes for completeness and clarity.
4. Approve the analytical results or recommend corrective action to the laboratory supervisor.

When a second trained analyst is not available a peer review is not completed.

**Supervisory Review:**

Following analyst and peer review the data is forwarded to the laboratory section supervisor for review. The supervisor will:

1. Review the data package for completeness and clarity.
2. Follow-up on the peer review recommendations.

Designated reviewers normally perform the peer and supervisory reviews for GC-MS data. The reviewers are identified on the organizational chart in Appendix A.

**Administrative Review:**

The results of all analyses are reviewed for compliance with quality control criteria and technical correctness before data is released to the Project Manager for distribution to clients. Designated reviewers in the Metals, Conventional and Organic laboratories perform administrative reviews. Personnel responsible for administrative reviews are noted in the Organizational Chart in Appendix A to this LQAP.



Administrative review is the final data validation process. Personnel performing the administrative review are responsible for the final sign-off and release of the data. Following administrative review the data is released to Project Managers for incorporation into the final data deliverable package.

Administrative review will:

1. Verify that the analytical package submitted for reporting is complete and contains all necessary information and documentation.
2. Verify that appropriate and necessary data qualifying flags (Listed in Appendix N) have been used.
3. Verify that method blank and LCS data are acceptable, quality control requirements were met for surrogates in all samples and blanks, and that all necessary re-analyses or dilutions were performed.
4. Check the technical validity (i.e. are total metal > dissolved metals, is the cation/anion balance correct, etc.) of the complete data set.
5. Verify that all necessary final data reports have been generated and that all necessary data and documentation are included in the package.
6. Approve data reports for release.

## **10.2 Quality Assurance Review**

10% (1 out each 10) final data packages are reviewed by ARI's QA staff for compliance with ARI's QA Program. This assessment includes, but is not limited to, review of the following areas:

1. Reporting and analysis requirements
2. Initial and continuing calibration records
3. Quality control sample results (method blank, LCS, spikes, replicates, reference materials)
4. Internal and surrogate standard results
5. Detection and reporting limits
6. Analyte identifications.





Data review activities are summarized and documented by the reviewer. The review notes are filed with the associated raw data in the project file. Any QA-related deficiencies identified during the data review will be forwarded to the QAPM for corrective action.



## SECTION 11: QUALITY CONTROL SAMPLE ANALYSIS AND EVALUATION

Routine analysis of quality control (QC) samples is necessary to validate the quality of data produced in the laboratory. ARI routinely analyses the following quality control samples: method blank (MB), holding blank (HB), laboratory control sample (LCS), matrix spike (MS) and sample replicate (MD or MSD). Section 11.3 defines these QC samples. The number and type of QC analyses depend on the analytical method and/or the QA/QC protocol required for the analyses. An expected result has been defined for each type of QC analysis. If quality control sample results meet all specified criteria, the analysis is considered to be “in-control” and the data acceptable. Conversely, quality control sample results that do not meet the specified criteria indicate that the procedure may not be generating acceptable data and corrective action may be necessary to bring the process “in-control”.

In addition to QC analyses, ARI routinely uses surrogate standards to measure the efficiency of all analyses targeting organic analytes.

Detailed information concerning sample preparation batches, QC analyses and surrogate standards follow:

### 11.1 Sample Preparation Batch

All QC samples will be associated with a discrete sample preparation batch. A preparation batch is defined as 20 or fewer field samples of similar matrix processed together by the same analysts, at the same time, following the same method and using the same lot of reagents. Additional batch requirements are detailed in ARI’s method specific standard operating procedures. Each preparation batch will be uniquely identified. All samples, field and QC, will be assigned an ARI LIMS ID number and will be linked to their respective preparation batch. Each sample batch will contain all required QC samples in addition to a maximum of twenty field samples.

ARI will accommodate client, QC protocol or QAPP specific sample batching schemes.



## 11.2 QC Sample Requirements

Each preparation batch will include, at a minimum, a method blank (MB) and a laboratory control sample (LCS). Additional QC samples will be analyzed based upon the specific QC protocol required, data deliverable requirements or client request. ARI recommends that QC samples used to measure analytical precision also be included in each sample batch. These may include: a matrix spike and a matrix spike duplicate pair; a sample duplicate and a matrix spike pair or an LCS duplicate (LCSD) for comparison with the LCS.

## 11.3 QC Sample Definitions

### 11.3.1 Method Blank (MB)

A method blank is an aliquot of water or solid sample matrix that is free of target analytes and is processed as part of a sample batch. The MB is used to verify that contaminants or compounds of interest have not been introduced into samples during laboratory processing. MBs will be spiked with surrogate standards for all organic analyses.

ARI defines an acceptable MB as one that contains no target analytes at a concentration greater than one-half ARI's reporting limit or 5% of an appropriate regulatory limit or 10% of the analyte concentration in the sample which ever is greatest.

A minimum of one method blank will be included in each preparation batch. A maximum of twenty samples may be associated with one method blank. An acceptable MB is required prior to analysis of field samples from a preparation batch. For methods not requiring pre-analysis sample preparation, a minimum of one method blank will be analyzed immediately prior to sample analysis, periodically throughout the analytical sequence, and also at the end of the sequence.

The results of the MB analysis will be reported with the sample results.

### 11.3.2 Holding Blank (HB)

Holding blanks are organic-free water samples that are placed in each volatile organic sample storage refrigerator to monitor for possible cross-contamination of samples within the storage units. A holding blank from each refrigerator will be analyzed every 14 days. Holding Blank



analyses will be reviewed by laboratory management and archived in ARI's electronic document archive.

### **11.3.3 Laboratory Control Sample (LCS)**

An LCS is processed as part of each preparation batch, and is used to determine method efficiency. An LCS is an aliquot of water or solid matrix free of target analytes to which selected target analytes are added in known quantities. The analytes spiked into LCS samples are listed in ARI's method specific SOPs. LCS will be spiked with surrogate standards for all organic analyses.

Following analysis the percent recovery of each added analyte is calculated and compared to historical control limits. Current control limits are listed in Appendix K of this document. When calculated recovery values for all spiked analytes are within specified limits, the analytical process is considered to be in control. Any recovery value not within specified limits requires corrective action prior to analysis of any field samples from the associated preparation batch.

A minimum of one LCS will be prepared for each sample preparation batch. LCS analysis for those methods not requiring pre-analysis sample preparation will be performed after each continuing calibration. The results of all LCS performed will be reported with the sample results. A maximum of twenty samples may be associated with one LCS.

Specific clients or QA protocol may require the analysis of a duplicate LCS. When LCS duplicates are analyzed the failure of any analyte in either LCS to meet QC limits must trigger a corrective action.

### **11.3.4 Replicate Analysis**

Replicate analyses are often used to determine method precision. Replicates are two or more identical analyses performed on subsamples of the same field sample at the same time. Replicate analyses should be performed on samples that are expected to contain measurable concentrations of target analytes.

The calculated percent difference between replicates must be within specified limits or corrective actions are required. Percent differences exceeding the specified limit signal the



need for procedure evaluation unless the excessive difference between the replicate samples is clearly matrix related.

For inorganic analyses, a minimum of one replicate set should be processed for each analytical batch. Replicate sample analyses are not routinely performed for organic parameters. Instead, analytical precision is evaluated through the analysis of a duplicate matrix spike sample (MSD).

In order to perform replicate analyses, ARI's must receive sufficient volume to prepare the replicate aliquots.

Field replicates submitted to the laboratory will be analyzed as discrete samples.

### **11.3.5 Matrix Spike**

A matrix spike is an environmental sample to which known quantities of selected target analytes have been added. The matrix spike is processed as part of an analytical batch and is used to measure the efficiency and accuracy of the analytical process for a particular sample matrix. The analytes spiked into MS samples are listed in ARI's method specific SOPs. MS samples will be spiked with surrogate standards for all organic analyses.

Following MS analysis the percent recovery of each spiked analyte is calculated and compared to historical control limits. If recovery values for the spiked compounds fall within specified limits, the analytical process is considered to be in control. When calculated recovery is outside of historical limits corrective action is recommended.

Matrix spike duplicate (MSD) analyses are often used to measure method precision and accuracy. In this case the relative percent difference for recovery of spiked compounds is calculated and compared to established criteria.

Unless directed otherwise, ARI's policy is to prepare a matrix spike and a duplicate with each batch of samples for inorganic analysis and an MS/MSD set for each batch of samples for organic analyses. Analyte recovery and RPD values are reported with sample data.



### **11.3.6 Standardized Reference Material (SRM)**

An SRM is material analyzed and certified by an outside organization to contain known quantities of selected target analytes independent of analytical method. SRMs are normally purchased from outside suppliers outside of ARI and are supplied with acceptance criteria. Analysis of SRM is used to assess the overall accuracy of ARI's analytical process. SRM are routinely analyzed with each batch of samples for wet chemistry (conventional analysis) samples. External reference samples are analyzed after instrument calibration and prior to sample analysis. Compound recovery values not within the specified limit signal the need to evaluate either the calibration standards or instrumentation.

### **11.3.7 Other Quality Indicators**

In addition to analyzing the quality control samples outlined previously, various indicators are added to environmental samples to measure the efficiency and accuracy of ARI's analytical process. Surrogate standards are added to extractable organic samples prior to extraction to monitor extraction efficiency. Surrogate standards will also be added to volatile organic samples prior to analysis to monitor purging efficiency. Internal standards are added to metals digestates for ICP-MS analyses and to organic samples or extracts prior to analysis to verify instrument operation.

The calculated recovery of surrogate analytes is compared to historical control limits to aid in assessing analytical efficiency for a given sample matrix.

## **11.4 Control Limits**

To provide a means for evaluating whether or not a process is in control, acceptance limits have been established. These are based on internal, historical data for organic analyses and method specified limits for inorganic analyses. Samples associated with a specific program or contract (such as the USEPA Contract Laboratory Program) will be evaluated against program/contract-specified criteria. Routine samples will be evaluated against internally generated control limits. Project specific control limits will be used as required provided they have been reviewed for feasibility and approved by laboratory management.

Results of QA analyses are transferred from the LIMS to a control limit and chart generation program. The QAPM coordinates control chart and control limit generation. Control limits will



be generated for LCS compound recoveries, surrogate recoveries, and matrix spike compound recoveries, on a method and matrix specific basis. Advisory control limits will be utilized for analyses performed on an infrequent basis until a sufficient number of usable data points are collected. Control limits are updated at least annually, but may be updated more frequently if method or instrument changes have been made. Laboratory control and acceptance limits are detailed in Appendix K.

Two levels of control limits are utilized in evaluating process control: warning limits and action limits. Limits are statistically determined from values obtained from LCSs or other control samples. Warning limits, within which 95% of all results are expected, equal  $\pm$  two standard deviations from the average result. Action limits, within which 99.7% of all results are expected, are equal to  $\pm$  three standard deviations from the average result. Mean values, warning limits, and action limits are necessary for thorough evaluation of process control.

### **11.5 Control Charts**

Control charts, in conjunction with other control sample analyses, are useful in verifying that an analytical procedure is performing as expected. The control chart provides a pictorial representation of how closely control sample results approximate expected values, as well as showing analytical trends. Indicated on the control chart are the mean and upper and lower warning and action limits. The warning and action limits are used to determine whether or not an analytical process is in control. The mean is used to determine whether results obtained for a procedure are trending upward or downward, which may ultimately affect the accuracy of sample results.

The QA Officer will coordinate generation of control charts based on laboratory data at least semi-annually. These control charts will be distributed to and reviewed by section supervisors and managers. Any significant trends or variations in results will be identified, and the source of the trend corrected. Copies of control charts will remain on file in the QA section. At the bench/instrument level, individual results from quality control samples are evaluated against the limits.



## **SECTION 12: CORRECTIVE ACTIONS AND REESTABLISHMENT OF CONTROL**

To produce quality data, it is important that all aspects of the analytical process are under control and that all specified quality control criteria are met. On occasion, however, procedures, reagents, standards, and instrumentation can fail to meet specified criteria. Should any of those situations occur, the quality of data produced may be compromised. When procedures no longer appear to be in control, sample processing will be halted and appropriate actions will be taken to identify and rectify any instrument malfunctions or process-related issues. Prior to resuming sample analysis, verification of control will be made through the analysis of various control samples. Actions taken and observations made during reestablishment of control will be fully documented on the bench sheet or as an Analyst Note. Only when control has been regained and all actions documented will sample processing resume. This ensures that no results generated during the suspect period will be reported.

### **12.1 Responsibilities**

It is the responsibility of all laboratory personnel involved with sample processing to be able to determine whether or not a procedure is in control and to verify that all data are produced under conditions that are “in control”. It is at the analytical level that unacceptable conditions are most easily detected and addressed. These personnel are also responsible for employing and documenting all necessary corrective actions taken to regain control of a procedure. Samples processed during suspect periods will be reprocessed, and suspect data will be appropriately annotated to indicate that it is of questionable quality. The analytical staff will verify that all data submitted for review has been generated under acceptable conditions. All anomalies will be documented on the Analyst Notes form and will include such information as: type and source of anomaly, reasons for the anomaly, and actions taken to correct the problem. All personnel involved with subsequent and final data review are responsible for verifying that data were generated under acceptable conditions. If suspect data are identified at the review level, responsible analysts should be contacted to determine whether additional actions (such as reanalysis) will be taken. In addition, reviewers will confirm that anomalies





noted by the analyst were indeed addressed and that appropriate corrective actions were taken.

On occasion, it is not possible to generate data that meet all Quality Control Standards. This may be due to sample volume limitations or sample matrix effects. It is the responsibility of the analytical and data review staff to document these situations and to maintain communication with the Project Management staff. The Project Management staff, in turn, is responsible for notifying the client or specifying additional actions to be taken. Project Managers are further responsible for ensuring that clients fully understand which data are questionable and the reasons why acceptable results could not be generated.

It is the responsibility of the QAPM to perform regular reviews of corrective action procedures to ensure that unacceptable conditions or suspect data will be identified prior to releasing results. Section managers and supervisors are responsible for ensuring that appropriate corrective action procedures are in place and that all staff members are trained to identify and act upon “out of control” situations.

## **12.2 Corrective Actions**

There are various stages of the analytical process where the procedure may fall out of control and require corrective action. In general, all procedures and equipment will be monitored to verify that control is maintained during sample processing. The following details those stages as well as the actions taken to reestablish and verify control.

### Sample Preparation

During sample preparation, all glassware associated with a specific sample will be clearly labeled to eliminate the possibility of sample mix-up or mislabeling. Laboratory staff will ensure that sample-identifying labels are accurately completed and that correct sample identification is maintained at all times. If a sample appears to have been misidentified or mixed with another sample during preparation, the suspect samples will be discarded and new aliquots taken. If there is insufficient sample for a second preparation, the situation will be documented on the bench sheet and the Project Manager will be immediately notified.

Addition of surrogate standards or matrix spiking solutions will be carefully monitored to ensure that all samples are accurately fortified. Volumes and standard solution numbers of all



standards added to samples will be recorded on the bench sheet. If there is suspicion that a sample has been incorrectly spiked a new sample aliquot should be prepared. If there is insufficient volume for re-preparation, the bench sheet will be annotated to indicate which samples may be inaccurately fortified.

If sample matrix hinders processing per standard procedures, the section supervisor or manager will be consulted for guidance on appropriate actions. Preparation of smaller sample aliquots or employment of different procedures may be necessary. Any deviations from normal protocols will be documented on the bench sheet.

If at any time during sample preparation sample integrity is compromised or a procedural error is noted, the sample will be discarded and re-prepared. If insufficient sample volume is available for re-preparation, the situation will be documented on the bench sheet and the Project Manager will be immediately notified.

### Calibration and Tuning

Prior to sample analysis, all instrumentation will be calibrated and tuned to ensure that equipment meets all criteria necessary for production of quality data. Equipment must meet the calibration criteria specified in the section entitled "Calibrations", per manufacturer specifications or per project/contract requirements. If these criteria are not met, corrective actions must be employed. Any corrective actions taken will be fully documented in the appropriate logbook, indicating the problem, the actions taken, and verification. Samples will not be analyzed until initial verification of system performance has been made. In the event that continuing calibration results do not meet criteria, sample analysis will not resume until corrective actions have been employed or the system has been re-calibrated.

GC/MS Analyses - Analysis of the instrument performance check solution (BFB or DFTPP) will meet the specified ion abundance criteria. Initial calibration standards at a minimum of five concentrations will meet specified response factor and percent relative standard deviation criteria. If criteria are not met for initial calibration, the system will be inspected for malfunction. The initial tuning and calibration will be repeated, with all necessary corrective actions taken, until calibration criteria are met.

A check of the calibration curve will be performed at a minimum of once every 12 hours. All response factor criteria will be met. Additionally, the percent difference between the initial and continuing calibrations will meet specified criteria. If criteria



are not met, the system will be inspected for malfunction. The initial tuning and calibration verification will be repeated, with all necessary corrective actions taken, until calibration criteria are met.

Internal standard responses and retention times for standards will meet specified criteria. Any sample not meeting internal standard criteria will be reanalyzed. If reanalysis yields the same response and the instrument is determined to be functioning correctly, the failure to meet criteria will be attributed to sample matrix interference. No further re-analyses will be required.

GC Analyses - Organochlorine pesticide calibrations will be evaluated using either USEPA CLP or SW-846 guidelines. The Resolution Check standard will meet resolution criteria and Endrin and DDT breakdown in the Performance Evaluation standard will meet breakdown criteria. Initial calibrations will meet percent relative standard deviation criteria. If, during the initial calibration sequence, criteria are not met, the system will be inspected for malfunction and the initial calibration be reanalyzed. Samples will not be analyzed until all initial calibration criteria are met.

Continuing calibrations of either the mid-level calibration standard or Performance Evaluation standard will be analyzed every 12 hours. If continuing calibration criteria are not met, the system will be inspected for malfunction and corrective actions will be taken to bring the system back into compliance. If, after corrective actions, the system is still not in compliance, re-calibration will be performed. After the system has been successfully corrected or re-calibrated, all samples previously analyzed between the acceptable and unacceptable continuing calibration will be reanalyzed.

If, during the analytical sequence, retention time shifting occurs, the system will be inspected for malfunction and corrective actions will be taken to bring the system back into compliance. If, after corrective actions, the system is still not in compliance, re-calibration will be performed. After the system has been successfully corrected or re-calibrated, all samples with retention times outside the specified windows will be reanalyzed.

For all other analyses, initial calibration standards analyzed at a minimum of five concentrations will meet percent relative standard deviation criteria. If criteria are not met for initial calibration, the system will be inspected for malfunction. The calibration will be repeated, with all necessary corrective actions taken, until calibration criteria are met.

A check of the calibration curve will be performed after every 10 samples. All percent differences between the initial and continuing calibrations will meet specified criteria. If criteria are not met, the system will be inspected for malfunction and re-calibration will be performed. Samples analyzed between an acceptable and unacceptable calibration check will be reanalyzed.

Metals and Inorganic Analyses - Initial calibrations will be verified by analyzing a calibration check standard immediately after calibration. The percent differences between the initial calibration and calibration check standard will meet specified percent difference criteria. If criteria are not met, the system will be inspected for



malfunction. The initial calibration and calibration check will be reanalyzed until acceptance criteria are met.

The calibration check standard analyzed after every 10 samples will meet percent difference criteria. If the calibration check standard is not acceptable, the system will be inspected for malfunction and re-calibration will be performed as necessary. Samples analyzed between acceptable and unacceptable calibration check standards will be reanalyzed.

### Instrument Blanks

Prior to sample analysis, instrument and/or calibration blanks may be evaluated for the presence of target analytes. If analytes are detected, the concentrations must be below the reporting limits for those analytes. If analytes are detected at levels above the reporting limits, the source of contamination will be identified. Sample analysis will not commence until analyte levels in instrument and calibration blanks are below the reporting limits. Instrument and calibration blanks are analyzed for VOA analysis only if sample carryover is suspected.

Instrument and calibration blanks will also be analyzed throughout the analytical sequence. These will not contain target analytes at levels above the method detection limits for organic parameters or the reporting limit for inorganic parameters. If one or more analytes exceed the RL, an additional blank will be analyzed. If analyte levels are still above the method detection limits, the system will be inspected for malfunctions and the source of contamination will be identified. Sample analysis will not resume until instrument and calibration blank analyte levels are below the RL. Organic samples analyzed between acceptable and unacceptable blanks will be evaluated to determine the need for reanalysis per the following guidelines:

If no target analytes are detected in the samples, reanalysis will not be required.

If sample target analyte levels are above the method detection limits, samples will be reanalyzed at analyst discretion. Reanalysis will be dependent upon the analyte levels and whether or not there is likelihood that analytes detected are a direct result of system contamination.

If the analytes present at unacceptable levels in the instrument blank are not of interest or concern in the associated samples, reanalysis will not be required. This is often a consideration for ICP analyses where analytes of concern may be only a subset of the possible analytes.

Methods for the analysis of inorganic analytes require that all samples associated with an out of control blank be re-analyzed.



## Method Blanks

Prior to sample analysis, method blanks will be evaluated for the presence of target analytes. Ideally, no target analytes should be present in the method blank. If analytes are detected at or above the Reporting Limit, the method blank will be reanalyzed to verify that the contamination is not a result of instrument carryover or malfunction. If the presence of target analytes is confirmed, the concentrations must be below the RL for those analytes.

Several volatile and semi-volatile compounds and certain elements are considered to be common laboratory contaminants. Concentrations of these common laboratory contaminants may exceed the method detection limits, but may not be present at concentrations greater than five times the method reporting limits. Target analytes considered to be common laboratory contaminants are:

### **Volatile Organic Compounds**

*Methylene Chloride*

*Acetone*

*2-Butanone*

### *Semi-volatile Compounds*

*Dimethylphthalate*

*Diethylphthalate*

*Di-n-butylphthalate*

*Butylbenzylphthalate*

*bis-(2-Ethylhexyl) phthalate*

*Di-n-octylphthalate*

If target analyte concentrations in the method blank exceed the acceptable levels and instrument malfunction or contamination has been ruled out, the method blank and all associated samples will be re-prepared and reanalyzed. If there is insufficient sample volume remaining for reprocessing, the Project Manager will be notified. If it is necessary to report results associated with an unacceptable method blank, the results will be qualified to indicate possible laboratory contamination.



In the event that an analyte detected in the samples  $\geq 20$  times the method blank levels re-preparation and reanalysis is not required. It is assumed that any contamination in the method blank is insignificant and will not affect final quantified results.

### Laboratory Control Samples

Prior to sample analysis, the laboratory control sample (LCS) will be evaluated to verify that recovery values for all spiked compounds are within the specified acceptance limits. If LCS recoveries are out of control, corrective action is required. Corrective actions may include anything from a written explanation in the case narrative up to re-preparation and reanalysis of the entire sample batch.

### Internal Standards

For volatile and semi-volatile organic analyses, internal standard results will be evaluated after each analytical run to verify that the values are within acceptance limits. Internal standard values will be within -50% to +100% of the internal standard values in the continuing calibration. If any internal standard does not meet the criteria, the system will be evaluated to confirm that all instrumentation is operating properly. The sample will then be reanalyzed. If the reanalysis results do not meet acceptance criteria, it will be assumed that the sample matrix is affecting internal standard values. Further reanalysis will not be required.

### Surrogate

Surrogate recovery values will be evaluated after each analytical run to verify that the values are within acceptance limits. If recovery values are outside acceptance limits, the system will be evaluated to confirm that all instrumentation is operating properly. Documentation and bench sheets will be reviewed to verify that the concentrations of surrogate spike solutions added are accurate. For extractable organic analysis, bench sheets will be reviewed to determine if any additional dilutions or concentrations were performed. Bench sheets will also be reviewed for any explanatory notes about the sample.

If no system documentation, solution preparation or spiking errors are identified, the following considerations will be made:



When a volatile organic surrogate recovery value is outside of acceptable limits, the sample will be reanalyzed. If the reanalysis results are within acceptance limits, it will be assumed that the initial analysis was in error. If the reanalysis results are not within acceptance limits, it will be assumed that sample matrix is affecting surrogate recovery. Further reanalysis will not be required.

For semi-volatile organic analysis, one acid and one base/neutral surrogate recovery may be outside acceptance limits with no corrective action required provided the recoveries are at least 10%. If more than one acid or base surrogate standard is outside acceptance limits, or if any surrogate recovery value is less than 10%, the sample will be re-extracted and reanalyzed. If the reanalysis results are not within acceptance limits, it will be assumed that sample matrix is affecting surrogate recovery assuming all other QC analyses are acceptable. Further reanalysis will not be required. *Matrix spikes will not be re-extracted for unacceptable surrogate recovery values.*

For other extractable organic analysis, if a surrogate recovery value is outside of acceptance limits, the data will be reviewed to determine if the unacceptable surrogate is a result of matrix effect. If matrix interference is determined, the sample will be re-extracted or if re-extraction is not deemed useful, fully documented in the analytical narrative associated with the analyses. If a surrogate recovery is too low, based on the opinion of the final QA Data Reviewer, the sample will be re-extracted and reanalyzed.

### Matrix Spikes

Matrix spikes will be evaluated to verify that recovery values for all spiked compounds are within the specified acceptance limits. If unacceptable results are obtained, the system will be evaluated to confirm that all instrumentation is operating properly. Documentation and bench sheets will be reviewed to verify that the concentrations of spike solutions added are accurate. Sample preparation bench sheets will be reviewed to determine if any additional dilutions or concentrations were performed. Bench sheets will also be reviewed for any explanatory notes about the sample.

If no system, documentation, solution preparation, or spiking errors are identified, the following considerations will be made:

#### Organic Analyses:

If a matrix spike recovery value is outside the acceptance limits, but the LCS meets recovery acceptance criteria, re-extraction will not be required. It will be assumed that the unacceptable recovery value is a result of matrix effect.



If both LCS and matrix spike recovery values are outside the acceptance limits, the sample batch will be re-extracted and reanalyzed. This indicates the possibility of a systematic error that may affect the accuracy of final results.

Inorganic analyses:

Matrix spikes with unacceptable recovery values will be re-prepared and reanalyzed. If the reanalysis results are not within acceptance limits, it will be assumed that the sample matrix is affecting the recovery values. Further reanalysis will not be required.

A post-digestion spike analysis will be performed for all metals analyses processed following EPA-CLP guidelines.

Sample and Matrix Spike Replicates

Sample and matrix spike replicates will be evaluated to verify that percent differences between the replicates are within acceptable limits. Percent differences for metals and inorganic sample replicates will be within  $\pm 20\%$ . When percent difference criteria are not met, the system will be evaluated to confirm that all instrumentation is operating properly. Documentation and bench sheets will be reviewed to verify that the concentrations of spike solutions added are accurate. Sample preparation bench sheets will be reviewed to determine if any additional dilutions or concentrations were performed. Bench sheets will also be reviewed for any explanatory notes about the sample.

If no system, documentation, solution preparation, or spiking errors are identified, the following considerations will be made:

If percent difference values between sample replicates for metals and inorganic analyses do not meet acceptance criteria the Project Manager in consultation with ARI's client will determine whether to re-analyze the samples or flag the analytical results. If the samples are reanalyzed and results are not within acceptance limits, it will be assumed that the sample is not homogeneous, causing the poor analytical precision. Further re-analyses will not be required.

Replicate sample analyses are not routinely performed for organic parameters.

If percent difference values between matrix spike replicates do not meet acceptance criteria, but spike recovery values are acceptable, no re-extraction or analysis will be required. It will be assumed that the sample is not homogeneous, causing the poor analytical precision.

If percent difference values between matrix spike replicates do not meet acceptance criteria and recovery values in one or both replicates are not acceptable, the sample and associated matrix spike replicates will be re-prepared and reanalyzed. If the





reanalysis results are not within acceptance limits, it will be assumed that the sample is not homogeneous, causing the poor analytical precision. Further re-analyses will not be required.

### Samples

In addition to monitoring sample quality control indicators, samples will be evaluated to determine the need for reanalysis. The following conditions will be considered while evaluating samples:

If a target analyte detected in a sample exceeds the upper limit of the instrument calibration range, the sample will be diluted and reanalyzed. Dilution and reanalysis will be performed until the analyte concentration falls within the linear range of calibration. If the sample requires dilution to such a level that surrogates are no longer detectable and analytical accuracy is questionable, the sample will be re-prepared using a smaller sample aliquot.

Samples will be evaluated for matrix interference that may affect analyte detection and quantification. Appropriate cleanup procedures will be employed to remove interference. Samples will be diluted and reanalyzed as required to minimize background interference. If it is not possible to remove all interference, reported results will be qualified as necessary.

If low-level analytes detected in a sample are suspected to be a result of instrument carryover, the sample will be reanalyzed. If analyte levels remain approximately the same the initial results will be considered valid. If analytes are not detected during reanalysis, it will be assumed that the initial detection was due to carryover, and the initial results will not be reported.

If an instrument malfunction or procedural error occurs during analysis, all affected samples will be reanalyzed. If the malfunction appears to be an isolated incident, it will not be necessary to inspect the analytical system. If the malfunction appears to be an ongoing problem, the system will be inspected and necessary maintenance/corrective actions will be taken prior to resuming analysis.

### Sample Storage Temperatures

Every sample storage unit's temperature will be evaluated at the beginning of each day. Temperatures will be between 2 and 6 °C for refrigerators and < -10 °C for freezers. If a temperature is outside the specified range, the unit's temperature will be adjusted to bring the temperature back within limits. The Temperature Log will be annotated to document the adjustment.

If adjustment does not bring the temperature within range, or if adjustment is not possible, the Laboratory Supervisor will be notified and will take corrective action. The Temperature Log will



be annotated to document the action. If the temperature fluctuation is chronic or extreme, the samples will be removed from the unit and placed in another storage unit until the malfunctioning unit is repaired or replaced.

### Balance Calibrations

Balances are serviced once a year by a certified technician. The service includes preventative maintenance and calibration.

Balance accuracy will be verified prior to balance use. The recorded weight will be within the acceptance criteria specified on the Calibration Log. If the recorded weight is not within the acceptance limits, the QAPM will be notified. The Calibration Log will be annotated to document the action. The balance will not be used until it can be verified that acceptance criteria can be met.

### Water Supply System

The water supply for the volatile organic and inorganic laboratories will be monitored daily for the presence of contaminants through the analysis of method and/or instrument blanks. Organic contaminants, especially chloroform, are early indicators of the need for preventative maintenance. If organic or other contaminants are detected, the system filters will be changed. After filters have been changed, an additional aliquot of water will be analyzed to confirm that contaminants are no longer present.

The water supply for the metals laboratory will be monitored daily. When the resistivity falls below 18 megaohm, system maintenance will be performed.



## Section 13: LABORATORY EVALUATION AND AUDITS

Routine evaluations of the laboratory ensure that all necessary quality control activities have been implemented and are being effectively utilized. It is the responsibility of the QAPM to ensure that quality control activities are periodically evaluated for compliance. Findings from these evaluations allow the laboratory to address and modify any procedures that are not in accordance with the laboratory Quality Assurance Program or accreditation program requirements.

A number of tools are available for monitoring laboratory performance. ARI evaluates the quality of laboratory performance through the use of

*Internal QA Audits*  
*Technical System Audits*  
*Data Quality Reviews*  
*Audits by Outside Agencies (External Audits)*  
*Performance Evaluation Analyses*

Each audit provides an objective evaluation of laboratory performance. All internal audits and reviews are conducted according to specified guidelines. In addition, a collective review of audit findings provides an overall evaluation of the laboratory. Deficiencies noted during the course of an audit or performance evaluation will be addressed, a root cause analysis performed, and appropriate corrective actions will be taken. Follow-up audits will be conducted to verify that corrective actions have been satisfactorily implemented.

### Internal QA Audits

The Quality Assurance Officer regularly evaluates quality control activities within the laboratory to verify accuracy and compliance. The QAPM or designee routinely audits the following activities:

*Balance verification records*  
*Sample storage cooler temperature records*  
*Oven, incubator and water bath temperature records*  
*Chain of Custody records*  
*Standard preparation records*



Checklists are utilized to ensure consistent and complete audits. The checklists are included in SOP 1005S. Internal QA audit results will be summarized and reported to both staff and management. Corrective actions will be initiated as necessary. A schedule of internal QA audits is provided in Appendix L.

#### Technical System Audits

An audit of technical systems within the laboratory will be conducted at least annually. The audit will focus on the quality control and data generation/collection systems. The QAPM will conduct the audit with assistance from section managers and data reviewers. This evaluation will address areas such as:

*Calibration records*

*Maintenance records*

*Control charts*

*Computer vs. hard copy data*

*Adherence to SOPs and methods*

*Support system records (DI water, balances, pipettes, etc.)*

In addition, audit results from the past year will be reviewed to verify that all necessary corrective actions have been addressed and implemented.

#### Audits by Outside Agencies (External Audits)

As a requirement for many accreditation programs, on-site review of laboratory facilities and operations are conducted by clients or other outside agencies. The laboratory may be periodically audited by the following agencies:

USEPA Contract Laboratory Program

State of Washington Department of Ecology

State of Washington Department of Health

US Naval Facilities Engineering Service Center (NFESC) (formerly known as NEESA)

Hazardous Waste Remedial Actions Program (HAZWRAP)

US Army Corps. of Engineers

External audits are beneficial in that they provide an independent evaluation of the laboratory without internal influence or bias. The laboratory will be available for evaluation



at the convenience of the auditing agency. Laboratory personnel will be available during the audit to address questions or provide information regarding laboratory procedures. All comments, deficiencies, and areas of potential improvement noted by the auditor will be reviewed, and appropriate corrective actions will be taken to resolve the noted issues. A listing of laboratory accreditations is included as Appendix M.

### Performance Evaluations

Performance Evaluation (PE) sample analysis is a means of evaluating individual performance as well as the overall analytical system. In addition to the external audit, PE sample (PES) analysis is a requirement of many certification and accreditation programs. The laboratory routinely participates in the following performance evaluation programs:

*Analytical Standards, Inc.(ASI) Performance Evaluation Studies*

*USEPA Water Pollution (WP) Performance Evaluation Studies (Commercial Supplier)*

*USEPA Water Supply (WS) Performance Evaluation Studies (Commercial Supplier)*

*USEPA Contract Laboratory Program Quarterly Performance Evaluations (as required)*

A PES is a sample containing specific analytes in concentrations unknown to analysts. Comparison of the laboratory result to the "true" value determines the accuracy of the reported result and indicates the laboratory's ability to perform a given analysis. These results are also used to verify individual analyst proficiency. The QAPM will periodically submit internal "blind" performance evaluation samples to the laboratory sections for analysis. Values obtained by the laboratory will be compared to expected or true values. Parameters with reported values outside of the specified acceptable ranges will be evaluated by the analytical staff to determine the source of error. All necessary corrective actions will then be documented and implemented.

### Quality Assurance Reports to Management and Staff

In order to ensure that laboratory managers are kept apprised of quality related activities and laboratory performance, a "Quality Assurance Report to Management" the QAPM will



be produced annually and distributed to ARI management. The report will, at a minimum include:

1. Information concerning current and ongoing internal and external audits
2. Status and results of current or ongoing internal or external proficiency analyses
3. Identification of Quality Control problems in the laboratory
4. Information on all ongoing Corrective Actions
5. Current status of external certifications
6. Current status of the Staff Training Program
7. Outline of new and/or future Quality Assurance Program initiatives

The QAPM is responsible for follow-up and resolution of any deficiencies discussed in the report. Unresolved issues will remain on subsequent reports until addressed. Information such as performance evaluation results and audit reports will be distributed to the laboratory staff.

The application of these combined activities provides comprehensive monitoring and assessment of laboratory performance, and ensures that all data produced by ARI will be of the highest possible quality.



## **Section 14: APPENDICES**

- A. Laboratory Organization and Key Personnel Resumes**
- B. Training and Demonstration of Proficiency**
- C. Laboratory Facilities**
- D. Laboratory Instrumentation**
- E. Standard Operating Procedures**
- F. Sample Collection Containers and Preservatives**
- G. Laboratory Workflow**
- H. Analytical Methods**
- I. Method Detection Limits and Reporting Limits**
- J. Tuning and Calibration Criteria, Volatile and Semi-volatile Organics**
- K. Quality Control Recovery Limits**
- L. Internal Audit Schedule**
- M. Laboratory Certification and Accreditation**
- N. Data Reporting Qualifiers**
- O. Personal Conduct Statement**
- P. QA Policies**
- Q. Modification to ARI's LQAP**



## **Appendix A**

# **Laboratory Organization Chart and Key Personnel Resumes**





## KEY PERSONNEL RESUMES

### Mark Weidner

Laboratory Director

#### Profile

Mr. Weidner co-founded Analytical Resources, Inc., along with Brian Bebee, Sue Dunnihoo and David Mitchell. Prior to his co-founding of ARI in 1985, Mr. Weidner was the Head Mass Spectroscopist at Michigan State University and an instructor at the Finnigan Institute. As Laboratory Director, Mr. Weidner is responsible for overall laboratory performance, as well as facility expansion and major purchasing. Mr. Weidner is intimately familiar with all operational and analytical aspects of ARI and initiated many of the procedures currently in use.

#### Education:

M.S., Medicinal Chemistry, Purdue University, W. Lafayette, IN (1978).

B.S., Biochemistry, Michigan State University, E. Lansing, MI (1975).

#### Experience:

Laboratory Director/Co-founder, Analytical Resources, Inc., Seattle, WA (1985 to present).

Senior Chemist, City of Seattle, Seattle, WA (1981 to 1985).

Instructor, Finnigan Institute, Cincinnati, OH (1979 to 1981).

Mass Spectroscopist, Michigan State University (1978 to 1979).



**Brian Bebee**

Laboratory Manager

Administrative Services Manager

Profile:

Mr. Bebee co-founded Analytical Resources, Inc., along with Mark Weidner, Sue Dunnihoo, and David Mitchell. Prior to his co-founding of ARI, Mr. Bebee had gained extensive GC/MS experience as a GC/MS Chemist at the Municipality of Metropolitan Seattle, (METRO). When he co-founded ARI in 1985, Mr. Bebee became the Organics Division Manager until 1993, when he assumed the position of Laboratory Manager. As Laboratory Manager, Mr. Bebee is responsible for the day to day flow of all laboratory operations, including personnel, instrument, and procedural concerns. He is also responsible for the direct supervision of the Volatile and Semivolatile Laboratories.

Education:

A.A., Oceanography, Marine Biology, Biology, Shoreline Community College (1973).

Experience:

Laboratory Manager, Analytical Resources, Inc., Seattle, WA (1987 to present).

Organics Division Manager/Co-founder, Analytical Resources, Inc., Seattle, WA (1985 to 1987).

GC/MS/DS Operator, Municipality of Metropolitan Seattle, Seattle, WA (1980 to 1985).

Senior Water Quality Technician, Municipality of Metropolitan Seattle (METRO), Seattle, WA (1976 to 1980).

Water Quality Technician, Municipality of Metropolitan Seattle (METRO), Seattle, WA (1973 to 1976)



## David Mitchell

### Quality Assurance Program Manager

#### Profile:

Mr. Mitchell co-founded Analytical Resources, Inc., along with Mark Weidner, Sue Dunnihoo, and Brian Bebee. Prior to his co-founding of ARI, Mr. Mitchell had gained extensive experience in the environmental chemistry field as Senior Chemist and Trace Organics Laboratory Supervisor at the Municipality of Metropolitan Seattle (METRO). His responsibilities include the management of ARI's Quality Assurance/Quality Control Program.

#### Education:

Graduate Work in Chemistry (Organic/Biological), University of Wyoming, Laramie, WY (1970 to 1974).

B.S., Chemistry, Upper Iowa College, Fayette, IA (1970).

#### Experience:

Quality Assurance Manager, Analytical Resources Inc., Seattle, WA (1998 to Present)

Client Services Manager, Analytical resources Inc., Seattle WA (1987 to 1998)

Vice President/Co-founder of Analytical Resources, Inc., Seattle, WA (1985 to 1987).

Senior Chemist, METRO Trace Organics Laboratory, Seattle, WA (1979 to 1985).

Research Associate, Northwestern University Medical School (1974 to 1979).



## Susan Dunninghoo

Computer Services Manager  
Administrative Services Manager

### Profile:

Ms. Dunninghoo co-founded Analytical Resources, Inc., along with Mark Weidner, Brian Bebee, and David Mitchell. Prior to her co-founding of ARI, Ms. Dunninghoo had gained extensive experience in the environmental chemistry field through her work at Laucks Testing Laboratories, the City of Tacoma, and the Municipality of Metropolitan Seattle (METRO). As Computer Services Manager, Ms. Dunninghoo is responsible for the supervision of the Computer Services Section. She is also responsible for LIMS administration, which includes testing the LIMS for data integrity, as well as ensuring that client deliverable requirements are met.

### Education

Graduate work in Chemistry, University of Washington.

B.A., Chemistry, Augsburg College, Minneapolis, MN (1976)

### Experience

Computer Services Manager/Secretary, Analytical Resources, Inc., Seattle, WA (1985 to present)

Chemist, Laucks Testing Laboratories, Seattle, WA (1983 to 1985)

Chemist, City of Tacoma, Plant II, Tacoma, WA (1982 to 1983)

GC/MS/DS Operator, METRO TPSS Lab, Seattle, WA (1980 to 1982)



## Jay Kuhn

### Inorganic Division Manager

#### Profile:

Mr. Kuhn oversees ARI's Inorganic Division, which includes the Metals Sample Preparation, Metals Analysis, and Conventional Wet Chemistry sections. He has extensive experience in the environmental chemistry field, with an emphasis in inorganic analyses. Mr. Kuhn is experienced with in-house and EPA standard methods and protocols, as well as the operation, maintenance, and repair of ICP-MS, ICAP, CVAA, and Graphite Furnace instruments.

#### Education

Graduate work in Environmental Chemistry, University of Washington, Seattle, WA.  
B.S. Chemistry, University of California at Santa Barbara (1980)

#### Experience

Inorganic Division Manager, Analytical Resources, Inc., Seattle, WA (1992 to present)  
Metals Division Manager, Analytical Resources, Inc., Seattle, WA (1990 to 1992)  
Research Technologist III and Laboratory Manager, UW College of Forest Resources  
Chemical Analysis Cost Center (1985-1990)  
Research Technologist, UW College of Forest Resources Chemical Analysis Cost Center  
(1981 to 1985)



# **Appendix B**

## **Training**



## Qualification Requirements

In addition to on-the-job training, ARI recommends a specific level of education and experience for the following positions:

### *GC/MS Laboratory Supervisor*

A Bachelor's degree in chemistry or scientific/engineering discipline, three years experience operating GC/MS systems and one year supervisory experience.

### *GC Laboratory Supervisor*

A Bachelor's degree in chemistry or scientific/engineering discipline, three years experience operating GC systems and one year supervisory experience.

### *Sample Preparation Laboratory Supervisor*

A Bachelor's degree in chemistry or scientific/engineering discipline, three years experience in organic sample preparation and one year supervisory experience.

### *Data Systems/LIMS Manager*

A Bachelor's degree with four or more computer-related courses and three years experience in systems management or programming. A minimum of one year experience with software utilized for laboratory report generation is also recommended.

### *Programmer Analyst*

A Bachelor's degree with four or more computer-related courses and two years experience in systems or application programming. A minimum of one year experience with software utilized for laboratory report generation is also recommended.

### *Quality Assurance Officer*

A Bachelor's degree in chemistry or a scientific/engineering discipline and three years of laboratory experience, including one year of applied experience with quality assurance.

### *Project Manager*

A Bachelor's degree in chemistry or a scientific/engineering discipline and three years of laboratory experience, including one year of applied experience with quality assurance.

### *GC/MS Chemist*

A Bachelor's degree in chemistry or a scientific/engineering discipline and at least one year experience operating a GC/MS system. Three years of GC/MS operations and spectral interpretation experience may be substituted in lieu of educational requirements.

### *Mass Spectral Interpretation Specialist*



A Bachelor's degree in chemistry or a scientific/engineering discipline and participation in training course(s) in mass spectral interpretation. Also, at least two years of experience in mass spectral interpretation is recommended.

*Purge and Trap Expert*

A Bachelor's degree in chemistry or a scientific/engineering discipline and one year experience operating a purge and trap type liquid concentrator interfaced to a GC/MS system.

*GC Chemist*

A Bachelor's degree in chemistry or a scientific/engineering discipline and at least one year experience operating a GC system. Three years of GC operations and maintenance experience may be substituted in lieu of educational requirements.

*Pesticide Analysis Expert*

A Bachelor's degree in chemistry or a scientific/engineering discipline and at least one year experience operating a GC system. Three years of GC operations and spectral interpretation experience may be substituted in lieu of educational requirements.

*ICP Spectroscopist*

A Bachelor's degree in chemistry or a scientific/engineering discipline and Four years of applied experience with ICP analysis of environmental samples. Four years of ICP experience may be substituted in lieu of educational requirements.

*ICP Operator*

A Bachelor's degree in chemistry or a scientific/engineering discipline and one year of experience operating and maintaining ICP instrumentation. Three years of ICP experience may be substituted in lieu of educational requirements.

*Atomic Absorption (AA) Operator*

A Bachelor's degree in chemistry or a scientific/engineering discipline and one year of experience operating and maintaining graphite furnace and cold vapor AA instrumentation. Three years of AA experience may be substituted in lieu of educational requirements.

*Conventional (Classical Chemistry) Analyst*

A Bachelor's degree in chemistry of a scientific/engineering discipline and one year of experience with classical chemistry procedures. Three years of classical chemistry experience may be substituted in lieu of educational requirements.

*Sample Preparation Expert*

A high school diploma and one college level course in chemistry. One year of experience in sample preparation is also recommended.





# **Appendix C**

## **Laboratory Facilities**



ANALYTICAL RESOURCES INC. occupies a total of 23,500 square feet of floor space located at 4611 S. 134<sup>th</sup> Place in Tukwila, Washington. The laboratory facility, constructed between September 2001 and June 2002, includes:

- State-of-the-art heating, ventilation and air conditioning (HVAC) systems to assure a clean comfortable working environment while maintaining air flow balance designed to minimize the possibility of sample cross contamination between laboratory areas.
- A central service area provides space for three walk-in coolers (356 sq. ft. total), two walk-in freezers (760 cubic ft.), eight reach-in freezers, and sample cooler storage.
- A data network linking all workstations to a centralized server room. All connections are made to managed switches and hubs and are protected by the latest firewall technology and uninterruptible power supplies.
- Distribution systems to deliver pressurized Air, Zero Grade Air, Argon, Helium, Hydrogen, Nitrogen and Argon/Hydrogen to the laboratory areas from a central location.
- A system to deliver ASTM Type 1 water directly to sinks in each laboratory area. Water is purified by filtration, ion exchange and reverse osmosis and continuously re-circulated through a filtration + ion exchange + UV radiation polishing loop that delivers water directly to the laboratories.
- An isolated and ventilated hazardous waste storage area.
- An electronic repair shop and storage room.
- Alarm monitored fire sprinkler and intrusion detection systems

The facilities are divided into five functionally-distinct sections as detailed below:

- 1) The Organics Division features three main laboratory areas as described below:
  - The Organics Extraction Laboratory (2400 sq. ft.) is utilized to isolate and concentrate organic compounds from various environmental sample matrices. The laboratory contains approximately 200 linear feet of bench space and nine fume hoods. It is equipped with two gel permeation chromatographs, an accelerated solvent extractor (ASE) and a gas chromatograph for extract screening purposes. The laboratory includes a separate area for extraction of aqueous samples, a glassware cleaning area and individual workstations for the laboratory supervisor and analyst.
  - The Semivolatile Organics Analysis Laboratory (3000 sq. ft) has 124 linear feet of instrument bench space plus personal workstations. The Laboratory is equipped with seven Gas Chromatographs (GCs) with six GC-MS instruments, one High Performance Liquid Chromatograph (HPLC) and a fume hood for preparation of standard solutions and dilution of samples. Each gas chromatograph is individually vented to the outside for removal of heat and potentially contaminated GC exhaust gases.
  - The Volatile Organics Analysis (VOA) Laboratory (2500 sq. ft) houses seven GC-MS and two GC-PID instruments dedicated to volatile organics analysis. Each instrument is vented to the outside. The laboratory area includes two fume hoods, a sample/standards preparation area, a TCLP preparation/tumbler room and sample holding refrigerators. The HVAC system maintains a positive air pressure in the laboratory using filtered air from outside of the building. This eliminates the possibility of cross contamination of samples with solvents from other areas of the laboratory.
- 2) The Inorganic Division includes a Trace Metals Laboratory and the Conventional Analyses Laboratory:



- Trace Metals Laboratory (3000 square feet)
    - The Metals Preparation Laboratory (1200 sq. ft) contains five fume hoods including two 8-foot polypropylene. An additional eight foot polypropylene laminar flow fume hood is housed in a separate class 1000 clean room. The lab is equipped with tumblers, hot-plates, digestion blocks, facilities for glassware cleaning, and a spectrophotometer for cold vapor analysis of mercury, a TCLP tumbler room, and storage areas.
    - The Metals Instrument Laboratory (1300 sq. ft) features two atomic absorption spectrometers for graphite furnace analyses, two inductively coupled argon plasma spectrometers (ICP) for simultaneous analysis of metals species, and an ICP-mass spectrometer for analysis of metals species at low detection levels.
    - A 500 sq. ft. Office provides desk area for Trace Metals laboratory personnel.
  - The Conventional Analyses (Wet Chemistry) Laboratory (2500 sq. ft.) contains approximately 200 linear feet of bench space, eight fume hoods and includes a separate microbiology room. Instruments in this lab include two Rapid-Flow Analyzers, two TOC analyzers, an ion chromatograph, two uv/visible spectrophotometers, and various other equipment necessary for the evaluation of inorganic parameters.
- 3) The Geotechnical Laboratory includes 2500 square feet of space with special areas and equipment for soil testing, treatability studies, and soil/sediment leaching studies. The Laboratory includes approximately 50 feet of linear bench space and 5 fume hoods.
- 4) The Sample Receiving Facility consists of an area to accept and log-in samples to ARI's Laboratory Information Management System (LIMS) and an area to prepare and ship sampling supplies.
- The Sample Receiving Facility (1000 sq. ft.) is equipped with two fume hoods, and 70 feet of bench space. Four computer terminals are available to log samples into ARI's LIMS.
  - The Sampling Containers Facility (500 sq. ft.) is used to prepare sampling containers for shipment to ARI's client designated locations.
- 4) Administrative Areas (8600 sq. ft.) include:
- The Quality Assurance Section
  - Executive Offices
  - Project Management Section
  - The Human Resources Section
  - The Computer Services Section
  - Two Conference Rooms
  - A Lunch Room
  - Several Storage Areas



## Appendix D

# Instrumentation and Computers

## **INSTRUMENTATION and COMPUTER SYSTEMS**

### **Organic Extractions Equipment**

**(ASE 1) Accelerated Solvent Extractor (1998)** – Dionex ASE 200

**(GPC 1) Gel Permeation Chromatograph (1985)** – Fluid Metering Inc. pump and ISCO UA-5 UV detector equipped with a 16 position autosampler used for clean-up of samples prior to final analysis.

**(GPC 2) Gel Permeation Chromatograph (2003)** – Fluid Metering Inc. pump and ISCO UA-5 UV detector equipped with a 16 position autosampler used for clean-up of samples prior to final analysis.

### **Gas Chromatograph - Mass Spectrometers (GC/MS)**

**(FINN I) Finnigan MAT Incos 50 (1993)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. System includes an HP 5890 GC, a Tekmar LSC 2000 Purge and Trap and a Delta Perspective PTA-30 autosampler for VOA analysis of either aqueous or solid samples.

**(FINN III) Finnigan MAT Incos 50 (1987)** - A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. System includes a Varian 3400 GC, a Tekmar LSC 2000 Purge & Trap and a Delta perspective PTA-30 autosampler for VOA analysis of aqueous samples.

**(FINN V) Finnigan MAT Incos 50 (1989)** - A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. System includes an HP 5890 GC, a Tekmar LSC 2000 Purge & Trap and a Delta Perspective PTA-30 autosampler for VOA analysis of either aqueous or solid samples.

**(NT I) Hewlett Packard (1994)** - A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. The system includes a Hewlett Packard 5890 Series II Plus GC, an HP 5972A MSD and a HP 7673 autosampler.

**(NT 2) Hewlett Packard (1999)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. System includes an HP 6890 GC, an HP 5973 MSD, an HP 7683 autosampler and an APEX Prosep 800 large volume injector.

**(NT3) Hewlett Packard (1999)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. System includes an HP 6890 Plus GC, an HP 5973 MSD, a Tekmar LSC 2000 Purge/Trap and a Dynatech Precision Sampling PTA 30 autosampler for VOA analysis of aqueous or solid samples.

## **Gas Chromatograph - Mass Spectrometers (GC/MS) (continued)**

**(NT4) Hewlett Packard (2001)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. The system includes an HP 6890-Plus GC, an HP 5973 MSD, an HP 6890 autosampler and an APEX Prosep 800 large volume injector.

**(NT5) Hewlett Packard (2002)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. The system is equipped with an HP 6890N GC, an HP 5973N MSD, a Tekmar LCS 2000 Purge and Trap and a Dynatech PTA 30 autosampler for VOA analysis of aqueous or solid samples.

**(NT6) Hewlett Packard (2002)** – A GC-MS system networked with a Hewlett Packard Unix Server running ThruPut Target 3.5 data analysis software. The system includes an HP 6890 Plus GC, an HP 5973 MSD and an HP 7683 autosampler.

## **Gas Chromatographs**

**Hewlett Packard 5890 Series II (2003)** – A GC system equipped with both FID and ECD detectors, capillary injectors, an autosampler and integrator. Used for screening samples before full extraction.

**(ECD 1) Hewlett Packard 5890 (1986)** - A GC system equipped with dual ECD detectors, two capillary injectors, a HP 7673A autosampler and ChromPerfect data system.

**(ECD 2) Hewlett Packard 5890 Series II (2003)** – A GC system equipped with dual ECD detectors, two Cool on column capillary injectors, an HP7673A autosampler and ChromPerfect data system.

**(ECD 3) Hewlett Packard 5890 Series II (1991)** – A GC system equipped with Dual ECD detectors, two Cool on column capillary injectors, an HP7673 autosampler and ChromPerfect data system.

**(ECD 4) Hewlett Packard 5890 Series II (1994)** – A GC system equipped with dual ECD detectors, a split/splitless capillary injector, HP7673 autosampler and ChromPerfect data system.

**(FID 2) Hewlett Packard 5890 (1987)** – A GC system equipped with an FID detector, a capillary injector, an HP 7673A autosampler and ChromPerfect data system.

**(FID 3 A, B) Hewlett Packard 6890 (1996)** – A GC system equipped with dual FID detectors, two capillary injectors, a dual tower HP 6890 autosampler, and HP Chemstation data system. A Restek GC Racer has been added to enhanced performance.

**(FID 4 A, B) Hewlett Packard 6890 (1996)** – A GC system equipped with dual FID detectors, two capillary injectors, a single tower HP 6890 autosampler, and HP Chemstation data system. A Restek GC Racer has been added to enhanced performance.

**(PID 1) Hewlett Packard 5890 (1988)** – A GC system equipped FID and PID detectors in series, an Dynatech PT30 autosampler and Tekmar LCS 2000 Sample Concentrator with ChromPerfect data system.

**(PID 2) Hewlett Packard 5890 – (1991)** –A GC system equipped with dual PID detectors, one in series with an FID, a Dynatech PT30 autosampler, an OI Analytical 4560 sample concentrator and a ChromPerfect data system.

**(ECD 5) Hewlett Packard 6890 Plus Micro – (2002)** – A GC system equipped with dual ECD detectors, two capillary column injectors, a dual tower HP 7683 autosampler, an APEX Prosep 800 large volume injector and an HP Chemstation data system.

**(FID 5) Hewlett Packard 5890 Series II (2005)** – A GC system equipped with FID and TCD detectors, an HP 7694 Headspace Sampler and HP Chem Station data acquisition system.

## **Inorganic Instrumentation**

**Perkin-Elmer SCIEX ELAN 6000 ICP-MS (1996)** - A completely automated ICP-Mass Spectrometer with autosampler and multitasking software. Computer controlled using ELAN NT Windows based software.

**Perkin-Elmer Optima 4300 ICP (2001)** - A completely automated dual view simultaneous ICP with auto-sampler and multitasking software.

**Varian 300Z (1992)** - A single channel atomic absorption graphite furnace instrument equipped with Zeeman background correction, and an auto-sampler.

**Varian 300Z (1991)** - A single channel atomic absorption graphite furnace instrument with Zeeman background correction, equipped with an auto-sampler.

**CETAC M-6000A Mercury Analyzer (2000)** – A fully automated high sensitivity cold vapor atomic absorption instrument dedicated to trace and ultratrace Mercury analysis. System is computer controlled with windows base software and an auto-sampler.

**Dionex Ion Chromatography DX 500 (1997)** - Fully automated system with an auto-sampler for quantitative anion analyses. The system is computer controlled using Peaknet software.

**Thermo Genesys 10 (2003)** - UV-VIS Spectrophotometer used for quantitative conventional analysis.

**Milton Roy 401 (1991)** - UV-VIS Spectrophotometer used for quantitative conventional analysis.



## **Inorganic Instrumentation (continued)**

**Alpkem RFA/2 Autoanalyzer (1990)** – The system is automated and computer controlled using Alpkem Soft Pac data acquisition for nutrient analysis.

**Lachat QuickChem 8000 Flow Injection Analyzer (2003)** – Automated flow injection instrument dedicated to low level nutrient analysis

**Dohrmann Apollo 9000 (2001)** - Total Organic Carbon (TOC) Analyzer. Includes an autosampler for water analysis

**Dohrmann DC190 TOC Analyzer with Boat Sampler (1994)** – Combustion/IR system dedicated to soil and sediment TOC analysis.

**Kontes Midi-Vap Cyanide Distillation Systems (1995)** – Each of the two systems is capable of simultaneously distilling up to 10 samples for cyanide analysis using small sample aliquots.

**Centrifuge (1987)** - Beckman Model GP with swinging bucket rotor and inserts for 250 ml bottles and scintillation vials

**Labconco 25 Place Block Digestion Unit.**

**Environmental Express Hot Block digestion blocks (8 ea) (1999-2002)** for digestion of samples prior to trace metals analysis.

**Hach COD Digestion Blocks (2)**

**Hach Ratio Nephelometer**

**Incubators: Lab-Line Ambi Hi-Lo Chamber and Thermolyne 41900.**

## **GeoTech Laboratory Equipment**

**Trautwein Soil Equipment 12 position flexible wall permeability station,**

**Soil Test Load frame, with 500, 2,000 and 10,000 pound load cells for QU, UU, and CU triaxial tests, with pore pressure.**

**Consolidation apparatus, 16 tsf**

**Geocon direct shear apparatus**

**Biosciences BI-1000, 8 position electrolytic respirometer**

**Microtox photo-luminescence toxicity tester**





**Beckman JP-21 refrigerated centrifuge with 6 x 500 ml fixed angle head**

**IEC DRP-6000 refrigerated centrifuge with a 4 x 1,000 ml swinging bucket head**

**Plas-Labs anaerobic test chambers**

**U.S. Army Corps of Engineers column settling; column and batch leaching apparatus**

## **Network Servers**

ARI's central laboratory computer is a Dell PC Server running the Windows NT platform. This system is home to ARI's Laboratory Information Management System (LIMS) database developed by Northwest Analytical of Portland, OR. The LIMS receives electronic data from all lab sections and produces hardcopy and electronic deliverables. In addition, the LIMS stores sample demographic data while providing a common tracking mechanism for all laboratory information.

The LIMS is connected to two sub-networks. Data is transferred electronically from the instrument data systems to the LIMS database. This key process enhances data integrity by reducing manual entry and manipulation of instrument output.

The metals section uses an Intel PC Server with the Windows 2000 Server operating system. This system runs as a file server for dBASE IV and MS Access 97 database applications. Once data is collected by the metals instrument computers, dBASE is used to process the data and transfer it to the LIMS database. The MS Access software has been customized by ARI's metals data supervisor to generate metals CLP forms and other internal reports.

The organics section uses an HP Unix Server with HP-UX 10.20 operating system. This system runs Target 3.4 data analysis software. All GC/MS and other GC instruments are networked to this system. In addition to providing one common platform for organics data processing, the Target software produces CLP forms for organics data packages.

The conventional analysis laboratory uses PC Workstations with MS Excel for data reduction. Data is manually entered into the LIMS systems using customized work lists.

Instrument data systems have been optimized with the latest software enhancements to reduce data processing time. Advances in processing have allowed ARI to meet the increasingly shorter turnaround requirements of our clients.

**Note: Extensive in-house replacement parts are available for lab instruments and computers, including spare circuit boards. A majority of all service maintenance is performed by ARI employees.**



# **Appendix E**

## **ARI Active SOPs**



<u>SOP #</u>	<u>Section</u>		<u>Version</u>	<u>Date</u>
<b>Sample Receiving / Project Management</b>				
001S	CSSR	Sample Receiving	019	7/13/05
003S	CSSR	Project Tracking	006	10/23/04
004S	CSPM	Data Storage, Archival and Retrieval	007	2/20/06
005S	CSPM	Project Management	004	10/30/04
0056S	CSPM	Handling of USDA Regulated Soil	001	10/12/05
<b>Computer Services</b>				
101S	CO	Software Quality Assurance (Draft)	003	2/25/04
<b>Data Reporting</b>				
201S	DS	GC-Data Reporting and Review	006	10/25/04
202S	DS	GC-MS Data Reporting and Review	004	10/25/04
203S	DS	Volatile Organics Data Reporting and Review	004	10/26/04
204S	DS	GC BETX Data Reporting and Review	004	10/26/04
205S	DS	Conventionals Data Review and Reporting	003	2/20/06
<b>Organic Extractions</b>				
300S	E	Sonicator Function Testing	007	10/14/05
301S	E	Organics Glassware Preparation	008	10/29/04
302S	E	Silica Gel Clean-up for Pesticides and PCB	002	4/24/06
303S	E	Tissue Extraction – Pesticide/PCB	001	8/4/05
304S	E	Soil Extraction – NWTPH-D, AK102, AK103 MicroTip Sonication	012	9/23/05
305S	E	BAN Extraction – Water – Separatory Funnel	013	11/01/04
306S	E	Gel Permeation Chromatography	003	10/28/04
308S	E	Water Extraction – NWTPH-D, AK102, AK103	013	5/17/05
311S	E	Pesticide/PCB Extraction – Water – Sep Funnel	017	4/27/06
315S	E	Butyl Tin Extraction – Soil/Sediment – Sonication	008	10/28/04
316S	E	Butyl Tin Extraction – Pore Water – Separatory Funnel	011	3/8/03
320S	E	Butyl Tin Species – Sediment – <i>in-situ</i> Ethylation	001	5/14/04
324S	E	Herbicide Extraction – Water – Separatory Funnel	012	7/18/06
325S	E	Herbicides Extraction – Soil – Macro-tip	010	2/23/06
326S	E	Extraction of Water for Organophosphorus Pesticides	008	8/11/06
327S	E	Extraction of Soil for Organophosphorus Pesticides	007	7/21/06
328S	E	Chlorinated Phenols – Water – Separatory Funnel	011	10/25/04
332S	E	PCB Extraction – Wipe Samples	008	3/8/03
333S	E	PCB Extraction – Soil - Medium Level	009	6/6/05
334S	E	Sulfur Removal from Sample Extracts	006	2/9/05
335S	E	Sulfuric Acid Clean-up of Sample Extracts	009	2/9/05
336S	E	Low Level Manchester Extraction for Pesticides and PCBs	014	1/31/05
340S	E	BAN Extraction – Tissue – Tissuemizer	009	7/28/05
341S	E	SIM-PNA Extraction – Water – Liquid Liquid	003	9/17/03



<u>SOP #</u>	<u>Section</u>		<u>Version</u>	<u>Date</u>
342S	E	Extraction of Soil Samples for NWTPH-HCID	009	3/9/05
344S	E	BAN Extraction – Water – Liquid-Liquid	011	8/15/05
349S	E	Paint Filter Liquids Test	006	10/26/04
350S	E	Pest/PCB Extraction – PSEP/PSDDA – Macro-tip	009	2/12/04
355S	E	SIM-PNA Extraction – Water – Separatory Funnel	003	9/17/03
357S	E	PNA Extraction – Soil – Micro-tip	002	10/28/04
359S	E	Sample Screening for PCB/ABN/PNA/PNA-SIM	006	10/27/04
360S	E	Extractions Opening/Closing Checklist	005	10/28/04
367S	E	Chlorinated Phenols – Soil – Micro-tip	003	5/2/05
374S	E	BAN Extraction – PSEP/PSDDA – Macro-tip	003	7/29/03
377S	E	BAN Extraction – Soil – Micro-tip	003	10/28/04
381S	E	Soil Extraction – NWTPH-D, AK102, AK103 – ASE	003	11/6/02
398S	E	EPH Extraction/Fractionation – Soil – Micro-tip	003	9/30/04
399S	E	EPH Extraction/Fractionation – Water	004	10/15/04
<b>Gas Chromatography</b>				
400S	GC	GC Analysis and General Operations	009	7/20/05
403S	GC	PCB Analysis – EPA Method 8082	015	9/25/05
404S	GC	Gasoline Analysis of Soil & Water (NWTPH-G)	010	10/9/04
405S	GC	Herbicides Analysis – EPA Method 8151	008	7/18/06
407S	GC	Diesel Hydrocarbon Analysis (NWTPH-D)	009	5/10/05
409S	GC	Hydrocarbon Identification (NWTPH-HCID)	006	12/14/05
410S	GC	BTEX Analysis by GC-PID – EPA Method 8021	009	10/6/04
412S	GC	Chlorinated Phenols – EPA Method 8040	003	10/30/03
421S	GC	Diesel & Residual Range Organics (AK102-103)	005	10/6/03
422S	GC	Gasoline Range Organics (AK101)	004	10/15/04
423S	GC	Pesticides Analysis – EPA Method 8081	010	9/23/05
425S	GC	PCB – Congener Analysis – GC-ECD	001	12/27/97
426S	GC	Glycol Analysis using GC-FID	004	9/10/03
427S	GC	Water Soluble SVOA via Direct Aqueous Injection	002	6/30/05
428S	GC	Extractable Petroleum Hydrocarbon	003	7/25/04
430S	GC	Volatile Petroleum Hydrocarbons	003	10/14/04
<b>Metals Sample Preparation and Analyses</b>				
500S	MP	Metals Glassware Prep.	003	10/18/04
502S	MI	Varian 300Z Graphite Furnace Analysis	008	10/22/04
505S	MP	Metals Sample Prep. Method 3020A (TWN)	008	9/22/04
506S	MP	Metals Sample Prep. Methods 7060A/7740 (RMA)	008	9/27/04
507S	MP	Metals Sample Prep. Method 3050B (SWC)	008	11/3/04
508S	MP	Metals Sample Prep. Method 3005A (RWC)	008	10/5/04
509S	MP	Metals Sample Prep. Method 3050B (SWN)	008	11/3/04
510S	MP	Metals Sample Prep. Method 3010A (TWC)	008	10/5/04
511S	MP	Metals Sample Prep. Method 7471 (SMM)	006	11/3/04
514S	MP	Metals Sample Prep. Filter/Wipe (PHN,PNM)	001	11/3/04
522S	MP	Metals Sample Prep. CLP Method 3005-M (RCN)	007	11/3/04
525S	MP	Metals Sample Prep. CLP Method 3005-M (RCC)	007	11/3/04



<u>SOP #</u>	<u>Section</u>		<u>Version</u>	<u>Date</u>
526S	MI	Metals Standards Prep. And Maintenance	006	10/20/04
527S	MI	Metals Spiking	008	10/20/04
529S	MP	Percent Solids Determination	004	11/3/04
531S	MP	TCLP Extraction: Method 1311	008	1/11/06
532S	MP	Metals Sample Prep. Method 7471A (SWM)	004	11/4/04
533S	MP	Metals Sample Prep. Method 7470A (TWM)	005	10/5/04
535S	MP	Metals Sample Prep. Method 200.8 (REC)	002	11/4/04
536S	MP	Metals Sample Prep. Method 200.8 (REN)	003	10/5/04
537S	MP	Metals Sample Prep. Method 200.8 (RHN)	003	10/20/04
538S	MI	Elan 6000 ICP-MS	005	2/5/04
539S	MI	Cetac Mercury Cold Vapor Analysis	002	10/20/04
540S	MI	ICP Analysis	004	10/20/04
<b>Wet Chemistry (Conventional) Analyses</b>				
600S	CV	Ferrous Iron	003	10/26/04
601S	CV	Cyanide	008	11/2/04
602S	CV	TOC – Soil and Sediment	008	3/1/03
603S	CV	Acidity	002	11/2/04
604S	CV	Alkalinity	003	3/18/04
605S	CV	Biochemical Oxygen demand	004	1/9/06
606S	CV	Bromide	002	11/2/04
607S	CV	Cation Exchange Capacity	003	11/2/04
608S	CV	Chlorophyll a	003	11/2/04
609S	CV	Chemical Oxygen Demand	002	11/2/04
610S	CV	Color (Visual Comparison)	003	11/2/04
611S	CV	Conductivity	003	11/2/04
612S	CV	Chloride (Automated)	003	11/2/04
614S	CV	Hexavalent Chromium	004	11/2/04
615S	CV	Ammonia (Automated)	005	4/21/04
616S	CV	Ammonia (ISE)	003	2/12/04
617S	CV	Nitrate & Nitrite+Nitrate	004	11/2/04
618S	CV	pH	005	2/23/06
620S	CV	Standards Preparation	003	11/2/04
621S	CV	Ion Chromatography	005	9/24/03
623S	CV	Fluoride	003	04/19/04
628S	CV	Microbiology (Coliform)	002	11/2/04
631S	CV	Phosphorus	003	10/3/03
632S	CV	Dissolved Oxygen	003	7/14/05
633S	CV	Phenol	004	10/27/04
634S	CV	Oxidation/Reduction Potential	003	11/2/04
635S	CV	Salinity	002	11/2/04
637S	CV	Sulfate (Automated)	006	11/2/04
639S	CV	Solids	005	11/2/04
640S	CV	Sulfide	002	7/14/05
641S	CV	Sulfite	002	11/2/04



<u>SOP #</u>	<u>Section</u>		<u>Version</u>	<u>Date</u>
642S	CV	Total Kjeldahl Nitrogen	002	7/14/05
643S	CV	Turbidity	002	11/2/04
645S	CV	Glassware Cleaning	002	11/2/04
648S	CV	Hexane Extractable Materials - EPA Method 1664	000	11/2/04
649S	CV	TOC-Aqueous	001	11/2/04
<b>Volatile Organic Analyses</b>				
700S	VOA	Volatile Organics Analysis – GC/MS	009	7/11/05
702S	VOA	GC/MS Volatiles – Autosampler Operation	004	11/1/04
703S	VOA	Volatile Organic Compounds by GC/MS SIM	(Draft)	
704S	VOA	Volatile Organic Standard Preparation	003	11/1/04
706S	VOA	Volatile Organic Analysis – EPA Method 524.2	005	2/23/04
707S	VOA	TCLP/ZHE Extraction for VOA	001	11/01/04
710S	VOA	GC Headspace Equilibrium Analysis for Methane, Ethane and Ethene	002	11/6/0
<b>Semi-Volatile Organic Analyses</b>				
801S	SVOA	PNA by GC/MS SIM	007	7/25/06
802S	SVOA	Butyl Tin Species (GC-MS-SIM)	009	5/17/04
803S	SVOA	Butyl Tin Species in Porewater (GC-MS-SIM)	007	1/10/01
804S	SVOA	Semivolatile Organics by GC/MS (8270D)	010	7/5/05
805S	SVOA	Organophosphorous Pesticides GC/MS-SIM	001	8/25/06
<b>Quality Assurance Procedures</b>				
1000S	QA	TCLP Extractor RPM Monitoring	004	7/29/05
1001S	QA	Refrigerator and Freezer Temperature Monitoring	010	8/17/05
1002S	QA	Laboratory Ethics	000	3/25/05
1003S	QA	Balance Monitoring	009	10/18/04
1004S	QA	Document Control – Lab. Forms and Logbooks	007	10/19/04
1005S	QA	Quality Assessment and Improvement	009	4/5/06
1006S	QA	Document Control–Standard Operating Procedures	005	9/16/05
1007S	QA	Internal Chain of Custody-Conventionals	005	10/23/04
1008S	QA	Internal Chain of Custody-Metals	005	10/18/04
1009S	QA	Internal Chain of Custody-SVOA	008	9/15/05
1010S	QA	Internal Chain of Custody-Volatiles	006	10/28/04
1012S	QA	Standard Preparation – GC and Semivolatiles	003	10/22/04
1013S	QA	Chemical Receiving and Reagent Preparation	005	5/23/05
1015S	QA	Pipette Verification	002	10/18/04
1016S	QA	Control Limits and Control Charts	003	10/19/05
1017S	QA	Training and Demonstration of Proficiency	006	9/13/05
1018S	QA	Determination of MDLs and RLs	005	09/11/03
1019S	QA	Chain of Custody, Archival & Disposal-Org. Ext.	003	10/20/04
1021S	QA	Manual Integration of Chromatographic Peaks	000	10/29/04
1022S	QA	Volumetric Ware Verification	001	10/15/04



## **Appendix F**

# **Sample Containers, Preservation and Holding Times**

### Summary of Sample Containers, Preservatives and Holding Time Requirements

Parameter	Method Reference	Container Water	Container Soil/ Sed.	Preservation	Holding Time Water	Holding Time Soil
Acidity	305.1/2310B	500 mL HDPE			14 Days	
Alkalinity	310.1/2320B	500 mL HDPE <sup>(1)</sup>			14 Days	
Ammonia	350.1/4500-NH3	500 mL HDPE	4 oz. WMG	2 mL (a)	28 Days	7 Days
Anions (Cl <sup>-</sup> , Br <sup>-</sup> , F <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , SO <sub>4</sub> <sup>-2</sup> , PO <sub>4</sub> <sup>-3</sup> )	300.0/9056	500 mL HDPE	4 oz. WMG		48 Hour	7 Days
BETX	8021/8260	2-40 mL vial <sup>(1)</sup>	2oz.WMGS <sup>(1)</sup>	(b)	14 Days 7 Days <sup>(2)</sup>	14 Days
Biological Oxygen Demand (BOD)	405.1/5210	1 Liter HDPE			48 Hours	
Butyl Tin Species	GC/MS (SIM)	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Cation Exchange Capacity	9080/MSA 8 & 9		4 oz. WMG			6 Months
Chemical Oxygen Demand (COD)	410.4/5220D	250 mL AG	4 oz. WMG	1 mL (a)	28 Days	28 Days
Chloride	325.2/325.3 300.0/9056 4500-CL	500 mL HDPE	4 oz. WMG		28 Days	28 Days
Chlorophyll a	SM10200H	1 Liter AHDPE			24 Hours	
Coliform, Fecal	SM9222D	Corning 4 oz.	4 oz. WMG	(d)	24 Hours	24 Hours
Coliform, Total	SM9222B/9132	Corning 4 oz.	4 oz. WMG	(d)	24 Hours	24 Hours
Color	110.2/2120B	500 mL HDPE			48 Hours	
Conductivity	120.1/9050A 2510B/MSA 10	500 mL HDPE	4 oz. WMG		28 Days	28 Days
Corrosivity	SM2330	500 mL HDPE			7 Days	
Cyanide, Total	335.2/9010B 4500-CN	500 mL HDPE	4 oz. WMG	2 mL (c)	14 Days	14 Days
Cyanide, Amenable	335.1/9010B 4500-CN G	500 mL HDPE	4 oz. WMG		48 Hours	14 Days
Cyanide, Weak Acid Dissociable (WAD)	SM4500 CN I	500 mL HDPE	4 oz. WMG	2 mL (c)	14 Days 48 Hours <sup>(2)</sup>	14 Days
Dissolved Oxygen	360.2/4500-O C	BOD bottle		fixed in field	8 Hours	
HEM / HEM-SGT	1664 / 9071	1 Liter AG	4 oz. WMG	5 mL (a)	28 Days	28 Days
FOG (Fats/Oils/Grease )	5520B/413.1	1 Liter AG	4 oz. WMG	5 mL (a)	28 Days	28 Days
Fecal Streptococci	SM9230C	Corning 4 oz.	4 oz. WMG	(d)	24 Hours	24 Hours
Fluoride	340.2/300.0 9214/9056 4500-F B	500 mL HDPE	4 oz. WMG		28 Days	28 Days
Herbicides	8151A	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Hardness (Calculation)	6010/2340B	500 mL HDPE		5 mL (f) <sup>(3)</sup>	6 Months	
Hexavalent Chromium (Cr <sup>+6</sup> )	7196A 3500 CR-D	500 mL HDPE	4 oz. WMG		24 Hours	28 Days
Iodide	345.1	500 mL HDPE			On Receipt	
Iron, Ferrous (Fe <sup>+2</sup> )	3500FE D	500 mL AG		2 mL (b)	On Receipt	
Metals	6010/7000/ 200.8 series	500 mL HDPE	4 oz. WMG	5 mL (f) <sup>(3)</sup>	6 Months	6 Months
Methane, Ethane, Ethene in Water	RSKSOP 175	4-40 mL vial <sup>(1)</sup>		(a)	14 days	
Mercury	7470/7471	500 mL HDPE	4 oz. WMG	5 mL (f) <sup>(3)</sup>	28 Days	28 Days
Nitrate	353.2/300.0 9056 4500-NO3 F	500 mL HDPE	4 oz. WMG		48 Hours	7 Days
Nitrate + Nitrite	353.2/300.0 9056 4500-NO3 F	500 mL HDPE	4 oz. WMG	2 mL (a)	28 Days 48 Hours <sup>(2)</sup>	7 Days





### Summary of Sample Containers, Preservatives and Holding Time Requirements

Parameter	Method Reference	Container Water	Container Soil/ Sed.	Preservation	Holding Time Water	Holding Time Soil
Oil & Grease (See FOG)						
Organophosphorous Pesticides	8141	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Pentachlorophenol	8041-M/8270D	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Pesticides/PCBs	8081A/8082	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Petroleum Hydrocarbon-Diesel (TPH-D) (DRO)	NWTPH-Dx AK102	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Petroleum Hydrocarbon-ID (HCID)	NWTPH-HCID 8015M	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Petroleum Hydrocarbons-Gas (TPH-G) (GRO)	NWTPH-G AK101	2 - 40 mL vial <sup>(1)</sup>	2oz.WMGS <sup>(1)</sup>	(b)	14 Days 7 Days <sup>(2)</sup>	14 Days
pH	150.1/9040B 9045C/4500-H+	500 mL HDPE	4 oz. WMG		24 Hours	14 Days
Phenols, GC/FID	8041M	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Phenols, Total	420.1/5530	500 mL HDPE	4 oz. WMG	2 ml (a)	28 Days	28 Days
Phosphorous, Total	365.2 4500-P B	500 mL HDPE	4 oz. WMG	2 ml (a)	28 Days	28 Days
Phosphorous, Ortho (Soluble Reactive Phosphorous – SRP)	365.2/300.0 9056/4500-P E	500 mL HDPE	4 oz. WMG		48 Hours	28 Days
Polynuclear Aromatic Hydrocarbon-PAH	8270D & SIM	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Salinity	SM2520B	500 mL HDPE			28 Days	
Semivolatile Organics	8270D	2-500 mL AG	8 oz. WMG		7 Days	14 Days
Solids, Total (TS)	160.3/2540 B	1 Liter HDPE	4 oz. WMG		7 Days	14 Days <sup>(5)</sup>
Solids, Total Suspended (TSS)	160.2/2540 D	1 Liter HDPE			7 Days	
Solids, Total Dissolved (TDS)	160.1/2540 C	1 Liter HDPE			7 Days	
Solids, Total Volatile (TVS)	160.4/2540 E	1 Liter HDPE	4 oz. WMG		7 Days	7 Days
Solids, Settleable (SS)	160.5/2540 F	1 Liter HDPE			48 Hours	
Solids, Volatile Suspended (TVSS)	SM2540E	1 Liter HDPE			7 Days	
Sulfate	375.2/300.0 9036/9056 4500-SO4F	500 mL HDPE	4 oz. WMG		28 Days	28 Days
Sulfide, Acid Volatile (AVS)	EPA 1991	500 mL HDPE <sup>(1)</sup>				14 Days
Sulfide	376.2 4500S2 D 9030B	500 mL HDPE <sup>(1)</sup>	2 oz.WMGS <sup>(1)</sup>	2 ml (e) + 1 mL (c) pH > 9.0	7 Days	7 Days
Sulfite	377.1 4500-SO3B	500 mL HDPE			24 Hours	
Total Kjeldahl Nitrogen (TKN)	351.2/351.3 4500-NORG	500 mL HDPE	4 oz. WMG	2 ml (a)	28 Days	28 Days
Total Organic Carbon (TOC)	415.1/5310B 9060/Plumb <sup>(4)</sup>	250 mL AG	4 oz. WMG	1 ml (a)	28 Days	28 Days
Turbidity	180.1/2130 B	500 mL HDPE			48 Hours	
Volatile Organic Compounds	524.2/624 8260/8260SIM	3-40 mL vial <sup>(1)</sup>	2 oz.WMGS <sup>(1)</sup>	(b)	14 Days 7 Days <sup>(2)</sup>	14 Days

**Containers:**

AG = Amber Glass Boston Round Bottle  
WMG = Wide Mouth Glass Jar  
WMGS = Wide Mouth Glass Jar with Septa  
HDPE = High Density Polypropylene  
AHPDE = Amber HPDE

**Preservation:**

(a) = 9N H<sub>2</sub>SO<sub>4</sub>  
HCl to pH<2.0  
(c) = 10N NaOH

(d) = Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (Sodium Bisulfite) Tablet

(e) = 2N ZnOAc

(f) = 1:1 HNO<sub>3</sub>

**Notes:**

(1) = No Headspace

(2) = When Unpreserved

(3) = Total Metals or field filtered samples only

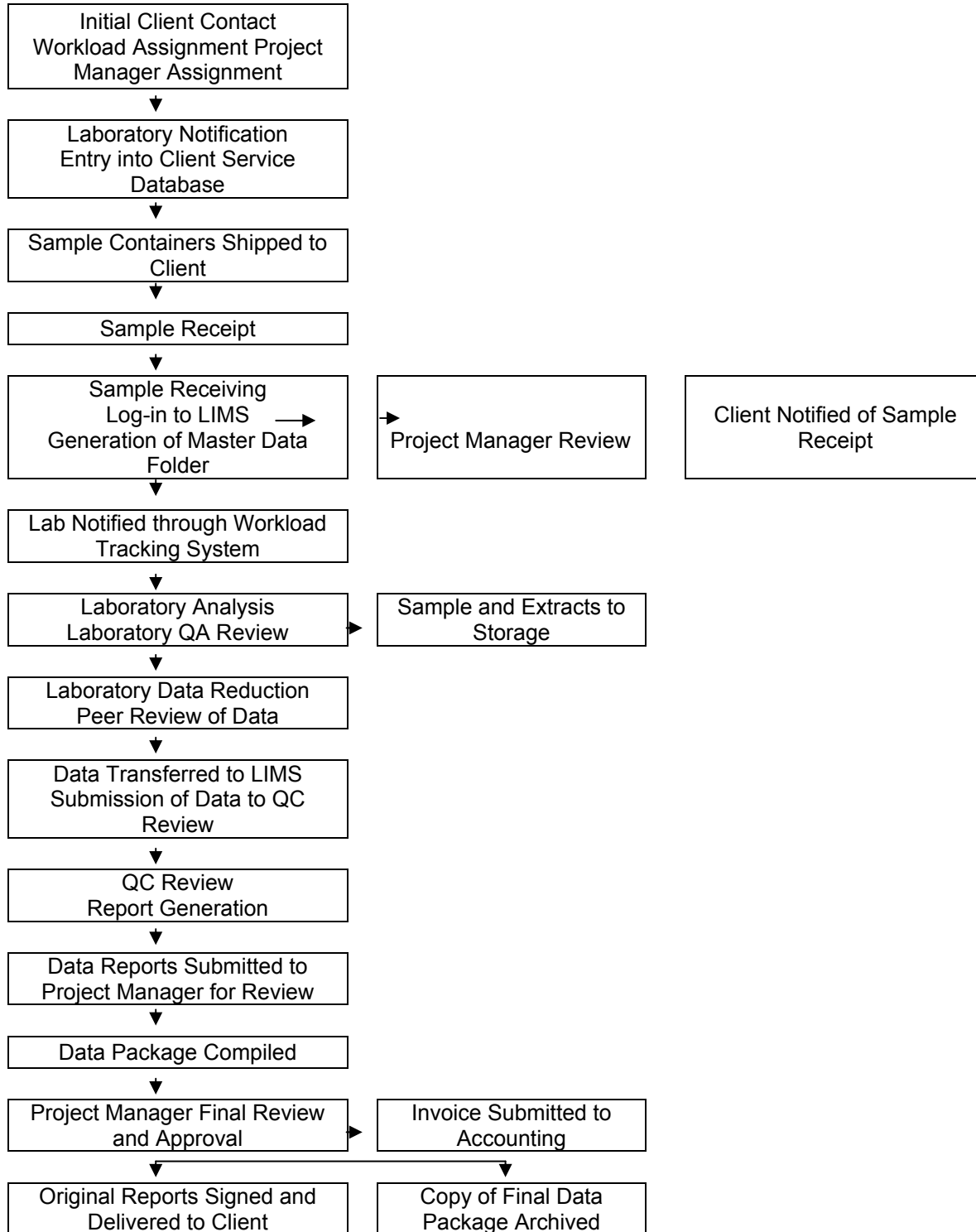
(4) = Plumb, R. H. Jr., *Procedures for Handling and Chemical Analysis of (b) (b) = Sediment & Water Samples*, May 1981, USACE Publication AD/A103788

(5) = When requested as a separate analyte. TS to correct for dry weight has the same holding time as the analytical parameter



# **Appendix G**

## **Laboratory Workflow**





# **Appendix H**

## **Analytical Methods**



## ORGANIC ANALYSES

Parameter	Methods	Technique
<b>Volatiles (GC/MS)</b>	524.2/624/8260B	GC/MS
	Low Level Vinyl Chloride & 1,1 – Dichloroethene	GC-MS-SIM
<b>Volatiles (GC)</b>		
Volatile Aromatics	602/8021B	GC/PID
<b>Semivolatiles (GC/MS)</b>		
Semivolatile Organics	625/8270D	GC/MS
Polynuclear Aromatic Hydrocarbons (PNA/PAH)	625/8270D	GC/MS (SIM)
Isotope Dilution Semivolatiles	1625	GC/MS
Butyl Tin Species	Krone (1988)	GC/MS-SIM
<b>Pesticides/GC Analyses</b>		
Chlorinated Pesticides	608/8081A	GC/ECD
Aroclors/PCBs	608/8082	GC/ECD
PCB Congeners	ARI Method	GC/ECD
Phenols	604/8041	GC/FID
Chlorinated Phenols	8041 (mod)	GC/ECD
Pentachlorophenol	8151A (mod)	GC/ECD
Organophosphorous Pesticides	614/8141A	GC/NPD
Polynuclear Aromatic Hydrocarbons (PNA/PAH)	610/8100	GC/FID
Chlorinated Hydrocarbons	612/8121	GC/ECD
Herbicides	615/8151A	GC/ECD
Glycols	ARI Method(SOP 426S R2)	GC/FID
Hydrocarbon ID	NWTPH-HCID	GC/FID
Gasoline Range Hydrocarbons	(N)WTPH-G/AK101/WI-GRO	GC/FID
Diesel Range Hydrocarbons	(NWTPH-D/AK102/WI-DRO)	GC/FID
Extractable Petroleum Hydrocarbons	ARI Method	GC/FID
Volatile Petroleum Hydrocarbons	ARI Method	GC/PID
<b>Organic Sample Preparation and Clean Up</b>		
TCLP / SPLP Extraction		1311 / 1312
Sonication		3550B
Soxhlet		3540C
Accelerated Solvent Extraction (ASE)		3545B
Separatory Funnel		3510C
Continuous Liquid-Liquid		3520C
Alumina Clean-up		3610B



Florisil Clean-up	3620B
Gel Permeation (GPC)	3640A
Silica Gel	3630C
Sulfur Clean-up	3660B
Sulfuric Acid Clean-up	3665A

## INORGANIC ANALYSES

Parameter	Methods	Technique
<b>Wet Chemistry</b>		
Acidity	2310/305.1	Titrimetric
Alkalinity	2320/310.1	Titrimetric
Ammonia	4500NH <sub>3</sub> H/350.1	Automated Phenate/ISE
Biological Oxygen Demand-BOD		
Carbonaceous – BOD	5210.B/405.1	5-day Winkler Titration
Bromide	4500Br.B	Phenol Red Colorimetric
Anions	300.0	Ion Chromatography
Cation Exchange Capacity	9080	Neutral Ammonium Acetate
Chemical Oxygen Demand	5220.D/410.4	Closed Reflux, Colorimetric
Chromium Hexavalent (Cr <sup>6+</sup> )	3500Cr-D/7196A	Diphenylcarbazide
Chloride	4500Cl.E/325.2	Automated Ferricyanide
Chlorophyll a	10200.H	Spectrophotometric
Coliform, Total / Fecal	9222.B/D	Membrane Filtration
Color	2120.B/110.2	Visual Comparison
Conductivity	2510/120.1	Electrometric
Corrosivity (CaCO <sub>3</sub> Saturation)	2330	Calc. (pH, Alk, TDS, Ca)
Cyanide, Total	4500CN.C/335.2/9010	PBA, Colorimetric
Cyanide, Amenable	4500CN.G/335.1	Alkaline Chlorination
Cyanide, WAD	4500CN.I	Weak Acid Distillation
Dissolved Oxygen	4500-O.C/360.2	Winkler Titration
Fats/Oils/Grease	5520.B/413.1/9070A	Gravimetric
Fluoride	4500F.C/340.2	Ion Specific Electrode
	300.0	Ion Chromatography
Formaldehyde	ASTM D-19 P216	Colorimetric
Hardness, Calculation	2340.B/6010B	Ca, Mg Calculation
Heterotrophic Plate Count	9215.D	Membrane Filtration
Iron (II) ferrous	3500Fe.D	Phenanthroline
Nitrate + Nitrite	4500NO <sub>3</sub> F/353.2	Automated Cd Reduction
Nitrate	4500NO <sub>3</sub> F/353.2	Calculated
	300.0	Ion Chromatography
Nitrite	4500NO <sub>3</sub> .F/353.2mod	Automated Colorimetric
	300.0	Ion Chromatography
Oil & Grease, Solids	5520.D/907	Gravimetric
Oil & Grease, Polar/Non Polar	5520.F	Gravimetric
PH	150.1	Electrometric
Phenols	5530.D/420.1/9065	4-AAP w/ Distillation
Phosphorous, Total	4500P.B/365.2	Colorimetric w/ digestion



Phosphorous, Ortho (SRP)	4500P.B/365.2 300.0	Colorimetric Ion Chromatography
Salinity	2520	Conductimetric
Silicate	4500Si.E/370.1	Heteropoly Blue
Total Kjeldahl Nitrogen (TKN)	4500N.org/351.4	Block Digest/ISE
Total Solids	2540.B/160.3	Gravimetric, 104°C
Total Suspended Solids (TSS)	2540.D.160.2	Gravimetric, 104°C
Total Dissolved Solids (TDS)	2540.C/160.1	Gravimetric, 180°C
Total Volatile Solids (TVS)	2540.E/160.4	Gravimetric, 550°C
Settleable Solids	2540.F	Volumetric
Streptococcus, Fecal	9230.C	Membrane Filtration
Sulfide	4500S <sup>2</sup> .E/376.1/9034	Iodometric
Sulfide, Low Level	4500S <sup>2</sup> .D/376.2	Methylene Blue
Sulfide, Acid Volatile	4500S <sup>2</sup> .D/376.2	Methylene Blue
Sulfate	4500SO <sub>4</sub> <sup>2</sup> .F/375.2/9036 300.0	Auto. Methylthymol Blue Ion Chromatography
Sulfite	4500SO <sub>3</sub> <sup>2</sup> .B.377.1	Iodometric
Total Organic Carbon (TOC)	5310.B415.1/PSEP	Combustion NDIR
Turbidity	2130.B/180.1	Nephelometric
Total Lipids in Tissue	Bligh & Dyer (mod)	Gravimetric

### Trace Metals Analyses

#### Inductively Coupled Plasma (ICP):

Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sn, Sr, Th, Ti, Tl, V, (Li, Th, U, W - special request only)	Zn200.7 / 6010B	ICP
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#### Graphite Furnace (GFAA):

Ag, As, Cd, Sb, Pb, Se, Tl	200 Series / 7000 Series	GFAA
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#### Cold Vapor (CVAA):

Hg	7470A/7471A	CVAA
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#### Inductively Coupled Plasma/Mass Spectroscopy (ICP-MS):

Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	200.8/ 6020 Mod.	ICP/MS
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### Trace Metals Sample Preparation

Toxicity Characteristic Leaching Procedure	1311
Synthetic Precipitation Leaching Procedure	1312
Digestion for Total Recoverable or Dissolved Metals	3005A
Digestion of Aqueous Samples for Total Metals by ICP	3010A
Digestion of Aqueous Samples for Total Metals by GFAA	3020A
Digestion of Sediment, Sludge and Soil	3050B



## Appendix I

### Method Detection Limits Reporting Limits

Summaries of method specific MDL studies and reporting limits are available on ARI's web site at:

<http://www.arilabs.com/portal/downloads/ARI-MDLs.zip>

MDL's and reporting are updated periodically. Assure that you have ARI's current detection limit data by downloading the files at the time of use.





## **Appendix J**

### **Quality Control Limits**

Method specific control limits are available on ARI's web site at:

<http://www.arilabs.com/portal/downloads/ARI-CLs.zip>

Control limits are updated periodically. Assure that you have ARI's current control limits by downloading the files at the time of use.



# **Appendix K**

## **Internal Audit Schedule**



## Schedule of Laboratory Quality Assurance Audits

<u>Process To Be Audited</u>	<u>Frequency</u>
Refrigerator/Freezer Temperature Logs	Monthly*
Oven/Incubator Temperature Logs	Monthly*
Balance Records	Quarterly*
Standard Records	Monthly#
Logbooks	Monthly#
SOPs	Monthly#
Chain of Custody	Monthly#
Internal Technical Systems	Annually
Post-Completion Project Review	Monthly**

\* all sections will be audited

# one section will be audited each month

\*\* frequency may be contract specific i.e. 10% of NFESC projects must be audited



# **Appendix L**

## **Laboratory Accreditations**



## **Laboratory Accreditations**

Analytical Resources Inc. is currently certified to perform environmental analysis by the State of Washington Department of Ecology, the State of Washington Department of Health and selected other states. ARI has also been accredited to perform various analyses for HAZWRAP (Hazardous Waste Remedial Actions Program), NEESA (Naval Energy and Environmental Support Activity), and the Navy Clean program.

ARI's laboratory QA/QC Program has been audited and approved by the USEPA Contract Laboratory Program for both organic and inorganic, the U.S. Army Corps of Engineers, The Boeing Company and Battelle Northwest Laboratories.

ARI analyzes performance evaluation samples quarterly for the EPA-CLP Program and semiannually for the EPA Water Pollution (WP) and Water System (WS) series. Also, all ARI laboratories periodically analyze blind in-house Performance Evaluation Samples as part of the laboratory QA/QC Program.

### **List of Accreditations**

- 1) State of Washington, Department of Ecology - Environmental Laboratory Accreditation Program
- 2) The Alaska State Department of Environmental Conservation - Laboratory Approval Program
- 3) United States Army Corps of Engineers (US ACOE)
- 4) United States Naval Facilities Engineering Service Center (NFESC) (formerly known as NEESA)

### **Continuing Contracts Resulting from On-Site Laboratory Audits**

- 1) The Boeing Company Corporate Environmental Affairs Division
- 2) Battelle Northwest Laboratories
- 3) The City of Seattle
- 4) The Port of Seattle



# **Appendix M**

## **Data Reporting Qualifiers**



## Data Reporting Qualifiers

Effective 12/28/04

### Inorganic Data

- U Indicates that the target analyte was not detected at the reported concentration
- \* Duplicate RPD is not within established control limits
- B Reported value is less than the CRDL but  $\geq$  the Reporting Limit
- N Matrix Spike recovery not within established control limits
- NA Not Applicable, analyte not spiked
- H The natural concentration of the spiked element is so much greater than the concentration spiked that an accurate determination of spike recovery is not possible
- L Analyte concentration is  $\leq 5$  times the Reporting Limit and the replicate control limit defaults to  $\pm 1$  RL instead of the normal 20% RPD

### Organic Data

- U Indicates that the target analyte was not detected at the reported concentration
- \* Flagged value is not within established control limits
- B Analyte detected in an associated Method Blank at a concentration greater than one-half of ARI's Reporting Limit or 5% of the regulatory limit or 5% of the analyte concentration in the sample.
- J Estimated concentration when the value is less than ARI's established reporting limits
- D The spiked compound was not detected due to sample extract dilution
- NR Spiked compound recovery is not reported due to chromatographic interference
- E Estimated concentration calculated for an analyte response above the valid instrument calibration range. A dilution is required to obtain an accurate quantification of the analyte.
- S Indicates an analyte response that has saturated the detector. The calculated concentration is not valid; a dilution is required to obtain valid quantification of the analyte
- NA The flagged analyte was not analyzed for



- NS The flagged analyte was not spiked into the sample
- M Estimated value for an analyte detected and confirmed by an analyst but with low spectral match parameters. This flag is used only for GC-MS analyses
- N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a “tentative identification”
- Y The analyte is not detected at or above the reported concentration. The reporting limit is raised due to chromatographic interference. The Y flag is equivalent to the U flag with a raised reporting limit.
- C The analyte was positively identified on only one of two chromatographic columns. Chromatographic interference prevented a positive identification on the second column
- P The analyte was detected on both chromatographic columns but the quantified values differ by  $\geq 40\%$  RPD with no obvious chromatographic interference

## Geotechnical Data

- A The total of all fines fractions. This flag is used to report total fines when only sieve analysis is requested and balances total grain size with sample weight.
- F Samples were frozen prior to particle size determination
- SM Sample matrix was not appropriate for the requested analysis. This normally refers to samples contaminated with an organic product that interferes with the sieving process and/or moisture content, porosity and saturation calculations
- SS Sample did not contain the proportion of “fines” required to perform the pipette portion of the grain size analysis
- W Weight of sample in some pipette aliquots was below the level required for accurate weighting





# **Appendix N**

## **Standards for Personal Conduct**



## Standards of Conduct

Since effective working relationships depend upon each of us, ARI expects certain minimum standards of personal conduct.

This list highlights general Company expectations and standards and does not include all possible offenses or types of conduct which may result in discipline or discharge. Management reserves the absolute right to determine the appropriate degree of discipline, including discharge, warranted in individual cases.

Employees engaged in the following activities, or similar activities deemed equally serious, will normally be terminated:

- theft or embezzlement
- disclosure of trade secrets or industrial espionage;
- willful violation of safety or security regulations;
- conviction of a felony;
- working for a competitor or establishing a competing business.

In addition, dismissal may result from other serious offenses such as:

- being intoxicated, under the influence or in possession of illegal drugs on the job;
- falsification of records;
- abuse, destruction, waste or unauthorized use of equipment, facilities or materials;
- gambling on the premises;
- chronic tardiness or absenteeism;
- insubordination;
- unwillingness to perform the job;
- unauthorized requisition of materials from vendors.

There may be no alcoholic beverages on the Company premises, other than at times designated as Company functions. At such times, non-alcoholic beverages will be provided as well.

Personal and corporate honesty and integrity have built the character of ARI. This good character is fundamental to our well-being, future growth and progress. It is vitally important that we avoid both the fact and the appearance of conflicts of personal interest with that of the firm, its clients, and any other professional contacts.

This policy requires that ARI employees have no relationships or engage in any activities that might impair their independence of judgment. Employees must not accept gifts, benefits, or hospitality that might tend to influence them in the performance of their duties. It is expected that there will be no employment by any competing company, nor any

employment by any outside interest or engagement in outside activity which might impair an employee's ability to render the full-time service to the company that employment involves.

If any possible conflict of interest situation arises, the individual concerned must make prior disclosure of the facts so that action may be taken to determine whether a problem exists and, if so, how best to eliminate it. Likewise, any financial interest in an organization doing business with ARI or which competes with us should be revealed to Company management. (Excluded from this requirement is ownership of securities traded in major stock exchanges or other recognized trading markets.)

Our standards are those generally expected of employees in any well-regarded, ethical business organization.

ARI further expects that each employee will:

- Be dressed and groomed appropriately for a business office. Employees in the laboratory areas are expected to dress in compliance with established safety procedures. Specific standards will be discussed with each employee during Health and Safety orientation. Your supervisor and the Administrative Services Manager always are available to answer questions.



## **Standards of Personnel Conduct – continued**

Maintain the confidential nature of Company information. Removal of Company documents, records, stored materials, computer printouts, or any similar information, or copies of such material or information from the office without specific permission is prohibited. Likewise, revealing confidential information to an unauthorized person or using such information in an unauthorized way is prohibited. If there could be any possible question about the applicability of this requirement to a given circumstance, ask your supervisor.

Use Company computer capabilities and facilities only for authorized business at authorized times and locations; observe strictly all computer security measures and precautions; enter, alter or delete no computer instructions or stored material apart from that required by faithful performance of assigned duties; remove, copy, use or permit to be used no computer software developed for, purchased by, or otherwise used by ARI except as required by faithful performance of assigned duties.

Conduct business dealings with clients and members of the public in a courteous manner.



# **Appendix O**

## **Quality Assurance Policies**



## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 1**

**SUBJECT: CORRECTIONS TO DATA/BENCHSHEETS**

**DATE: 8/2/96**

Manual corrections made on any raw data, bench sheet, logbook or document used during sample processing will be made in the following manner:

1. Draw a single line through the information to be deleted or corrected. The original information must remain readable.
2. Enter any new information, preferably above the original information.
3. Initial and date the correction.



## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 2**

**SUBJECT: LINING OUT UNUSED BENCHSHEET PORTIONS**

**DATE: 8/2/96**

All unused portions of logbook pages and benchsheets will be lined through so that information cannot be added at a later date. This will be completed in the following manner:

1. Line out unused portions of a logbook page or benchsheet by drawing a single line or "Z" through the unused portions.
2. Initial and date the page beside the lineout.
3. Do not line out a page or section until it is certain that no additional information will be added to the unused portions.



## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 3**

**SUBJECT: STOP WORK ORDERS**

**DATE: 8/28/96**

It is the responsibility of all staff members to address situations that may require the issuance of a "stop work order". Potential and actual "stop work orders" will be handled as follows:

1. If an analyst or technician observes a situation which will or may have a negative impact on data quality, that person will notify her/his section supervisor immediately.
2. The section supervisor will assess the situation. If it appears that a "stop work order" may be required, the section supervisor will notify the appropriate manager (inorganic or organic).
3. The supervisor and manager will then decide if a "stop work order" should be issued. The manager will make a final decision on whether or not to issue a "stop work order". The incident will be reported to the Quality Assurance Program Manager using a Corrective Action Request form.
4. If a "stop work order" is issued, the manager will inform the Project Managers and the QA section. The section supervisor will notify section staff of the order.
5. The laboratory manager involved will oversee the development and implementation of a Corrective Action Plan (CAP). Upon completion of the CAP the "stop work order" may be rescinded.
6. Prior to rescinding a "stop work order", verification must be made that control has been regained and that work may begin. Only the inorganic or organic manager may rescind a "stop work order".
7. When the "stop work order" is rescinded, the Project Managers, analytical staff and QA section will be notified. The QA section will require documentation verifying that the procedure is back in control.



## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 4**

**SUBJECT: SOP Review**

**DATE: 9/3/96**

All Standard Operating Procedure (SOP) documents will be reviewed and updated at least annually by qualified staff members. Laboratory management will review and approve all modifications to the SOPs.





## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 5**

**SUBJECT: Reporting Dilutions**

**DATE: 9/11/96**

Dilution factors will be recorded as whole numbers followed by "X" (i.e., 5X, 10X, etc.). This reporting convention will be used on run logs, bench sheets, raw data and final reports for all diluted samples, extracts or digestates or standards.



## QUALITY ASSURANCE POLICY

**POLICY NUMBER: 6**

**SUBJECT: Formatting for SOPs – Computer Related**

**DATE: 1/31/00**

Conventions for formatting computer-related instructions in SOPs

Commands should be indented and formatted as **courier** and one or two font sizes smaller:

```
USE PARAMS ORDER PARAMS  
BROW
```

Many systems and languages are *case-sensitive*, and case should match the syntax and/or stylistic standards of the language.

If only one command, like ***SET CENTURY ON***, is needed, it can be included in the rest of the text, so long as it is also italicized.

If the user must substitute a particular value in place of a general descriptor, italicize the descriptor, make it lowercase, and *do not make it bold*:

```
USE PARAMS ORDER PARAMS  
COPY TO TEMPARM FOR JOB = 'job' .AND. SAMPLE = 'sample'
```

In general, keywords, variable names, formatting codes, and descriptors should be in *courier* and *italicized*.



## QUALITY ASSURANCE POLICY

<b>POLICY NUMBER:</b>	<b>7</b>
<b>SUBJECT:</b>	<b>Manual Adjustment of Data</b>
<b>DATE of IMPLEMENTATION:</b>	<b>1/1/01</b>

Modern chromatographic instruments include computer software to identify a detector response as a chromatographic peak, characterize that peak and determine the relative height or area of the signal. The software utilizes parameters (threshold, slope, etc) that are adjusted by the instrument operator to optimize the results.

A single set of operator controlled settings that determine peak characteristics for an entire data file is defined as an "automated procedure". An automated procedure often characterizes chromatographic peaks incorrectly. ARI requires that trained analysts identify and resolve these errors using an alternate automated procedure or a "manual adjustment" of the data. Manual adjustment is defined as the process used by an analyst to adjust an individual peak or a subset of data in a chromatographic file.

1. The settings for a routine automated procedure normally used to process chromatographic data must be described in the method Standard Operating Procedure (SOP).
2. Trained analysts may substitute one automated procedure for another in order to optimize peak characteristics. The use of an alternate automated procedure must be permanently documented using either a software generated log file or analyst notes.
3. Manual adjustment of chromatographic peak characteristics will be used to correct the results of an automated procedure that, in a trained analyst's opinion, are clearly incorrect and will result in erroneous peak identification, integration or quantification.
4. Manual adjustment will be implemented in a reasonable and consistent manner. Guidelines for performing manual adjustment will be documented in method SOPs.
5. All manually adjusted data will be clearly identified for approval in the data review process. A permanent record of all manual adjustments will be maintained in both electronic and hardcopy versions of the raw data.
6. Manual adjustment of chromatographic files will not be used to falsify data for any purpose. Falsification of data through the use of manual peak adjustment is unethical, unlawful and will result in termination of the offending analyst.

Approval:

Quality Assurance Program Manager

Date



## QUALITY ASSURANCE POLICY

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<b>POLICY NUMBER:</b>	<b>8</b>
<b>SUBJECT:</b>	<b>Performance Evaluation Samples</b>
<b>IMPLEMENTATION DATE:</b>	<b>1/1/01</b>

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Performance Evaluation Samples (PES) will be analyzed on a periodic basis to monitor laboratory performance and/or meet the requirements of an external accreditation program. PES samples contain target analytes in concentrations unknown to laboratory personnel. PES may be submitted by a third party or prepared internally under the direction of ARI's QA personnel.

PES will be submitted blind to the laboratory whenever possible.

PES will be logged-in, prepared, analyzed and reported as a routine sample without special consideration.



## QUALITY ASSURANCE POLICY

<b>POLICY NUMBER:</b>	<b>9</b>
<b>SUBJECT:</b>	<b>Modifications to Analytical Methods Procedures or Reports</b>
<b>DATE of IMPLEMENTATION:</b>	<b>8/24/05</b>

This Policy defines the processes used to initiate and validate modifications to analytical processes, QA/QC protocol, data processing programs and algorithms, data reporting formats or other changes to analytical procedures or SOPs at Analytical Resources Inc. (ARI). The procedures outlined will also be used to validate project specific changes to analytical protocol and new analytical methods.

Changes to analytical procedures must be approved by ARI's Management (Managers and/or Supervisors) and be well documented using the following procedure:

1. Modification may be requested by any staff member. The modification must be requested using ARI's Corrective Actions Tracking System. Corrective Action requests for changes to analytical protocol or reports will assigned to the appropriate manager or supervisor by the initiator. As an alternative the request may be assigned to the QA Section. The Corrective Actions assignee may approve the project or re-assign the request for approval to a third party. The QA Section will monitor the progress of all requests.
2. The requestor must detail and justify the proposed modifications or additions when initiating a Corrective Action issue. Modifications must be approved by ARI management prior to any work performed to establish the modification.
3. The following must be in place before final approval and/or implementation of the proposed modification.
  - A. A new or revised SOP as appropriate including the modification or new protocol.
  - B. An Initial Demonstration of Proficiency as defined in ARI SOP 1018S for new or modified analytical procedures.
  - C. An MDL study following the procedure in ARI SOP 1018S for new or modified analytical procedure.
  - D. When appropriate, successful analysis of a blind Performance Evaluation Sample using new or modified procedures or data processing protocol.
  - E. Documentation that new or modified software provides the desired result.
4. ARI staff must have sufficient training to implement the procedural changes.
5. Notification of the modifications must be distributed to all affected personnel including appropriate client personnel.

## QUALITY ASSURANCE POLICY

<b>POLICY NUMBER:</b>	<b>10</b>
<b>SUBJECT:</b>	<b>Reporting of Target and Spiked Analytes For Dual Column GC Analyses</b>
<b>DATE of IMPLEMENTATION:</b>	<b>8/24/05</b>

Analytical Resources Inc. uses single injection, dual column gas chromatographs to simultaneously identify and confirm the presence of target or spiked analytes in some GC analyses. Only one quantitative value is reported for each target or spiked analyte. ARI's policy for deciding which value to report is outlined as follows:

1. ARI considers each column equally valid for compound identification and quantification. Both GC columns must be compliant with all quality assurance parameters outlined in ARI's SOPs and LQAP. Both GC columns must produce valid initial and continuing calibrations using the same calibration model.
2. The analytical value reported will be determined by comparison of the quantitative results of confirmed analytes as follows.

- a. The relative percent difference (RPD) between the results on the two columns ( $R_1$  &  $R_2$ ) is calculated using the formula:

$$RPD = \frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} \times 100$$

- b. If the RPD is less than 40% the greater of the two values is reported for both target analytes and spiked compounds. When required by specific QA protocol, by contract or client request the lower value will be reported for target analytes.
- c. If the RPD is greater than 40%, ARI's analyst must examine the chromatogram for anomalies (overlapping peaks, incorrect integration, negative peaks) and either correct the anomalies (i.e. perform manual integrations) or report the most appropriate target analyte value. The higher value will be reported for spiked analytes. ARI's analyst must provide a written evaluation of all analyses where an RPD exceeds 40% and this information must be passed on to ARI's client or the data user.



## **Appendix P**

### **Modifications to the LQAP**



## Modifications to ARI's LQAP

New Revision	Date	Modifications
12-008		<ol style="list-style-type: none"> <li>1. Added Methane, Ethane &amp; Ethene Info to Appendix F Table</li> <li>2. Updated SOP Table in Appendix E</li> </ol>
12-007	4/11/06	<ol style="list-style-type: none"> <li>1. Removed Appendix J – Tuning Criteria are in the SOP</li> <li>2. Changed BOD RL from 1 to 2 ppm</li> <li>3. Integrated all SVOA Soil/Sediment MDLs into One Table</li> <li>4. Added SIM Analysis to Soil/Sediment SVOA MDL Table</li> <li>5. Added SIM Analysis to Water SVOA MDL Table</li> <li>6. Updated MDL for SVOA in Water</li> <li>7. Updated MDLV for Pesticides in Soil (25g to 5mL)</li> <li>8. Updated MDLV for Pesticides in Soil (12g to 4mL)</li> <li>9. Updated MDLV for PCB in Water (500 to 1mL)</li> <li>10. Updated MDLV for PCB in Water (500 to 5mL)</li> <li>11. Updated MDLV for Chlorinated Phenols in Water (500 to 50mL)</li> <li>12. Removed Appendix I – MDL &amp; RL Summaries</li> <li>13. Updated MDL for SIM-PNA</li> <li>14. Updated MDLV for SIM-PNA</li> <li>15. Removed Appendix K – Control Limits</li> </ol>
12-006	1/16/06	<ol style="list-style-type: none"> <li>1. Updated MDL for TBT in Pore Water</li> <li>2. Updated MDL and MDLV for Toxaphene in Soil/Sediment</li> <li>3. Updated MDLV for VOA 8260B 20 mL Purge</li> <li>4. Added IDL, MDL &amp; RL for Low RL Mercury</li> <li>5. Updated all Metals MDL Verifications</li> <li>6. Updated MDLV for Water VOA using 5 mL purge</li> <li>7. Updated MDLV for PCB in Soil with Soxhlet Extraction</li> <li>8. Updated MDLV for SVOA (8270D) Analysis of Water using SepFunnel</li> <li>9. Updated MDL for GC-MS-SIM Analysis of Skydrol &amp; BHT in Water</li> <li>10. Updated MDL for Chlorophenols (8041) in Soil</li> <li>11. Modified RL for Chlorophenols in Soil &amp; Tissue</li> <li>12. Added Headspace GC (FID5) to Instrument List</li> <li>13. Updated Footnotes on Glycols RL Table</li> <li>14. Modified RL for 1,4-Dioxane in Water Method 8270D</li> <li>15. Updated MDL for Analysis of Soil for VOA</li> <li>16. Updated MDL for Analysis of Soil for JP-8</li> <li>17. Updated MDL for Analysis of Sediment for TBT</li> <li>18. Updated MDLV for Analysis of TBT in Water and Tissue</li> <li>19. Added MDL for Analysis of PCB in Tissue with 4 ppb RL</li> <li>20. Updated MDLV for PCB Analysis of Soil (Soxhlet) and Tissue (4 ppb)</li> <li>21. Updated MDLV for Manchester Analysis of PCB in Water</li> <li>22. Updated MDLV for Analysis of Gasoline in Soil and Water</li> <li>23. Updated MDLV for Analysis of BTEX in Soil and Water</li> <li>23. Updated MDLV for Analysis of Motor Oil in Soil and Water</li> <li>24. Updated MDLV for Analysis of VOA-SIM in Water</li> <li>25. Updated MDLV for Analysis of VOA (20 mL) in Water</li> <li>26. Updated MDL Table for Conventional</li> <li>27. Updated MDLV for Pesticides in Water (500 to .5 mL)</li> <li>28. Updated MDLV for PCB Analysis of Soil</li> <li>29. Updated MDLV for Chlorophenols (8041) in Soil</li> <li>30. Updated MDLV for JP4 in Water and Soil</li> <li>31. Updated MDLV for JP8 in Soil</li> <li>32. Updated MDLV for VOA (8260B) in Water 5 mL &amp; 20 mL Purge Volumes</li> <li>33. Updated MDL for PCB in Soil – Standard Analysis &amp; Medium Level</li> <li>34. Updated MDL for Pesticides in Water – Standard Analysis</li> <li>35. Updated MDL for SVOA in Water – Liq-Liq Extraction</li> <li>36. Updated MDLV for Chlorophenols in Water</li> </ol>





12-005	10/24/05	<ol style="list-style-type: none"><li>1. Added MDL for Chlorinated Phenol Analysis of Tissue (Method 8041)</li><li>2. Modified QA Policy 10</li><li>3. Established Implementation Date for QA Policies 09 &amp; 10</li><li>4. Updated MDLV for TBT in Water</li><li>5. Corrected MDL Value for bis-(2-Ethylhexyl)-phthalate in SVOA Tissue</li><li>6. Updated MDL for Pesticides in Soil</li><li>7. Modified Title Format of Selected MDL Tables</li><li>8. References to 8270 or 8270C changed to 8270D</li><li>9. Deleted MDL Tables for SVOA Analyses of Tissue</li><li>10. Updated MDLs for SIM-PNA in Water (SepFunnel) and Soil</li><li>11. Updated MDLV for Metals</li><li>12. Updated MDLV for Manchester Pesticides</li><li>13. Updated MDLV for TPH-D In Soil</li><li>14. Updated MDLV for SIM-PNA in Water with Liq-Liq Extraction</li><li>15. Updated MDLV for JP-4 in Soil</li><li>16. Updated MDLV for VOA Water 5 mL Purge</li><li>17. Corrected MTCA RL for Methoxychlor &amp; Manchester RL for all Pesticides</li><li>18. Updated MDL for Manchester Beta-BHC to reflect latest MDLV</li><li>19. Corrected Tissue Pesticide RLs</li><li>20. Updated MDLV for LVI-SIM-PNA in Water with Liq-Liq Extraction</li><li>21. Updated MDL for VOA-SIM Analysis of Aqueous Samples</li><li>22. Updated MDLV for PCB in Water (500 to 5 mL)</li><li>23. Updated MDLV for Diesel in Water (NWTPH-D &amp; AK102)</li><li>24. Updated MDLV for Chlorophenols in Aqueous Samples</li><li>25. Updated MDLV for Chlorophenols in Tissue Samples</li><li>26. Removed &amp; Archived Modifications to LQAP for 2002 &amp; 2003</li><li>27. Updated MDL for Skydrol/BHT Analysis in Water Using 8270-SIM</li><li>28. Removed Direct Aqueous Injection MDLs RL Table.</li><li>29. Updated SOP Table (Appendix E)</li></ol>
12-004	8/19/05	<ol style="list-style-type: none"><li>1. Added "A" Flag for GeoTech to Appendix N.</li><li>2. Updated MDL for JP-4 in Soil</li><li>3. Updated MDL for Pesticides in Tissue</li><li>4. Updated MDLV for JP-4 in Soil</li><li>5. Updated MDLV for Pesticides in Soil</li><li>6. Updated MDLV for Pesticides in Water</li><li>7. Updated MDLV for PCB in Soil (25g to 1 mL)</li><li>8. Updated MDLV for PCB in Water (500 to 5 mL)</li><li>9. Updated MDLV for TPH-D in Water</li><li>10. Updated MDLV for PNA-SIM in Water (Liq-Liq Extraction)</li><li>11. Updated MDLV for VOA in Water (5 mL 8260B)</li><li>12. Updated MDLV for VOA in Water (20 mL 8260B)</li><li>13. Updated MDL for PSSDA SVOA in Sediment</li><li>14. Updated Appendix E – SOP List</li><li>15. Corrected MDL for Pesticides in Soil Information (IA-80 not GU-32)</li><li>16. Corrected Reporting Limits for TBT in Water, Sediment &amp; Tissue</li><li>17. Added Control Limits for 1,4-Dioxane to SVOA List</li><li>18. Added low level RLs for BTEX Compounds</li><li>19. Updated MDLV for TBT in Pore Water</li><li>20. Updated MDLV for BTEX Water &amp; Soil</li><li>21. Updated MDLV for TPH-G in Water &amp; Soil</li><li>22. Updated Appendix E SOP Table</li><li>23. Updated MDLV for Motor Oil in Soil Using ASE</li><li>24. Updated MDLV for Motor Oil in Soil Using MicroTip</li><li>25. Updated MDLV for Motor Oil in Water Using SepFunnel</li><li>26. Updated MDLV for JP-4 in Water Using SepFunnel</li></ol>
12-003	7/15/05	<ol style="list-style-type: none"><li>1. Added MDLV for 5 mL VOA Analysis of Water – Method 8260B</li><li>2. Updated MDL for MTCA PCB in Water Samples</li></ol>



		<ol style="list-style-type: none"> <li>3. Added MDL for Soxhlet Extraction of PCBs</li> <li>4. Removed Aroclor 1242 from MDL Table</li> <li>5. Control Limits for HEM Changed to Equal Those in SOP 648S</li> <li>6. Updated MDL for PSDDA PCB Analysis.</li> <li>7. Added MDL for TBT in Tissue</li> <li>8. Updated MDL for 20 mL 8260B</li> <li>9. Updated MDLV for SIM-VOA</li> <li>10. Updated MDL for Pesticides in Soil</li> <li>11. Updated MDLV for TPH-D in Soil</li> <li>12. Added MDLV for PSEP Level Pesticides in Sediment</li> <li>13. Updated (added missing compounds) PSDDA SVOA MDLs</li> <li>14. Updated &amp; Corrected Appendix F (Containers &amp; Preservatives)</li> <li>15. Added "A" Flag for GeoTech to Appendix N.</li> </ol>
12-002	6/9/05	<ol style="list-style-type: none"> <li>1. Updated Motor Oil MDL (NWTPH-Dext &amp; AK103) for Soil</li> <li>2. Documented MDLV for Gasoline in Soil (Methods NWTPH-G &amp; AK101)</li> <li>3. Corrected units for DRO &amp; RRO MDL for water from mg/kg to mg/L</li> <li>4. Added MDL for JP-4 in Water using Sep Funnel Extraction</li> <li>5. Updated MDL for Sediment Analysis (Krone) of TBT using Sonication</li> <li>6. Updated MDL for SVOA Water SepFunnel</li> <li>7. Noted that BTEX –SIM MDL in Table was Medium Level Extraction</li> <li>8. Added MDL Verification Information for ICP Metals</li> <li>9. Updated MDL for TBT in Water and Pore Water – SepFunnel</li> <li>10. Updated MDLV for TPH-D Water – SepFunnel</li> <li>11. Added EPH and VPH RL Tables</li> <li>12. Added MDLV for JP-4 Analysis of Water – Sep Funnel</li> <li>13. Added MDLV for BTEX analysis of Soil</li> <li>14. Added MDLV for SVOA Water - SepFunnel</li> <li>15. Added MDLV for TBT Sediment</li> <li>16. Updated MDL for PSEP Pesticides in Sediment/Soil</li> <li>17. Updated MDL for Chlorinated Phenols in Water</li> <li>18. Updated MDL for Pesticides in Water – SepFunnel</li> <li>19. Added MDLV for 524.5</li> <li>20. Added MDLV for Metals</li> <li>21. Updated MDL for Manchester Pesticides</li> <li>22. Added Appendices to the Table of Contents</li> <li>23. Added MDL for PCB Analysis of Tissue</li> </ol>
12-001	4/5/05	<ol style="list-style-type: none"> <li>1. List of SOPs (Appendix E) Modified &amp; Updated as Appropriate</li> <li>2. MDL Verification for DRO in Soil Added</li> <li>3. MDL Verification for PCB Water Standard Analysis (HO-24) Added</li> <li>4. AK-101 Removed from BTEX MDL Table for Water</li> <li>5. Metals IDLs &amp; MDLs Updated</li> <li>6. BTEX MDL for Analysis of Water and Soil Updated</li> <li>7. RL for 1,4-Dioxane in SVOA Analysis of Water Changed from 1.0 to 5.0</li> <li>8. Control Limits for BTEX and Gasoline updated</li> <li>9. MDL for Gasoline in Soil Updated</li> <li>10. MDL for Diesel and Motor Oil in Soil Updated.</li> <li>11. Split TPH-G Table into Aqueous and Soil Table &amp; added MDLV for Water</li> <li>12. Entered updated MDLs for SIM-LVI-PNA</li> <li>13. Changed RL for 20 mL 1,2-Dibromo-3-Chloropropane from 2 to 0.5 ppb</li> <li>14. Updated MDLs for 524.2</li> <li>15. Updated Conventional MDLs</li> <li>16. Updated MDLs for 5 mL VOA analysis of Water Samples (8260B)</li> <li>17. Modified MDL Table for TPH-D Analysis of Water</li> <li>18. Updated TPH-D and TPH-Dext MDL for Water Analyses.</li> <li>19. Removed EPH and VPH MDLs from the LQAP</li> </ol>
11-028	12/31/04	<ol style="list-style-type: none"> <li>1. Modified definition of "Y" flag in Appendix N</li> <li>2. Updated MDL for TPH-D Soil</li> </ol>



		3. Updated Appendix M - Laboratory Certification and Accreditation
11-027	12/15/04	<ol style="list-style-type: none"> <li>1. Updated SOP List in Appendix E.</li> <li>2. Added AK-101 to BTEX/GRO Control Limit Table.</li> <li>3. Lowered RL for Benzene in MDL Summary for Method 8021B</li> <li>4. Added Additional Surrogates to VOA-SIM BTEX Control Limit Table</li> <li>5. Corrected BTEX MDLs for 8260-SIM to Reflect Sample Conc. Not On-Column values</li> <li>6. Updated SOP Table in Appendix E</li> <li>7. Modified VOA 5 mL Water RLs - Acrylonitrile &amp; 1,2,3-Trichloropropane</li> <li>8. Modified VOA mL Soil RL – 4-Methyl-2-Pentanone</li> <li>9. Corrected MDL Value for Methoxychlor in PSDDA Sediment Analysis.</li> <li>10. Modified definition of “Y” Flag in Appendix N</li> <li>11. Updated MDL for BTEX Water PID-2</li> <li>12. Updated MDL for Pesticides MTCA Analysis of Water</li> <li>13. Updated MDL for PSDDA SVOA Analysis</li> <li>14. Updated MDL for VOA Soil</li> <li>15. Updated MDL for SVOA, Water, Liq-Liq</li> <li>16. Updated MDL for Various PCB (1660) Analyses</li> <li>17. Updated MDL for TPH-G – Water &amp; Soil</li> <li>18. Updated MDL for SVOA Soil Micro Sonication</li> <li>19. Added MDL for Manchester Aroclor 1254</li> <li>20. Modified Control Limits for EPH Analyses</li> <li>21. Deleted MDL Table for SVOA, Soil, MacroTip Extraction</li> <li>22. Deleted MDL for Soil Skydrol/BHT, GC-MS-SIM</li> <li>23. Updated Instrumentation Listing (Appendix D)</li> </ol>
11-026	11/02/04	<ol style="list-style-type: none"> <li>1. Updated Control Limits for SIM-PNA</li> <li>2. Added Control Limit Table for Full Scan PNA Analysis (Method 8270D)</li> <li>3. Updated SIM-PNA Water MDL for NT-1</li> <li>4. Updated Appendix E – SOPs</li> <li>5. Modified PCB MDL Table –Remove Manchester &amp; Combine PSEP/Low Level Sediment MDLs</li> <li>6. Updated MDL for VOA SIM Water NT3</li> <li>7. Updated MDL Table for SIM Skydrol/BHT in Water</li> <li>8. Updated SOP Table in Appendix E.</li> </ol>
11-025	9/16/04	<ol style="list-style-type: none"> <li>1. Added new Appendix N listing Data Qualifiers &amp; changed designations for Appendices N, O &amp; P to O,P &amp; Q respectively</li> <li>2. Updated MDL Table for PCB Analyses.</li> <li>3. Combined MDL tables for SVOA Water &amp; Deleted Sep Funnel Table</li> <li>4. Updated PCB &amp; TPH-D MDL Tables</li> <li>5. Updated Equipment List (Appendix D) &amp; added GeoTech Equipment</li> <li>6. Revised MDL Table for FID Analysis of Polar SVOA (EPA Method 8015)</li> <li>7. Updated MDLs for Pesticide analysis of soil.</li> <li>8. Sediment Pesticide MDLs added to Soil Table, Sediment Table Deleted</li> <li>9. Control Limit for MS Recovery of Pyrene in Sediment Corrected</li> <li>10. Updated Cyclohexanone MDL (Finn 1, 20 mL purge)</li> <li>11. Updated SIM-PNA Soil MDL for NT-1</li> <li>12. Edited MDL Tables for SVOA for consistency and accuracy</li> <li>13. Modified EPH Reporting Limits</li> <li>14. Revised formatting on most MDL tables.</li> <li>15. Corrected dates for VOA Control Limit data</li> <li>16. Deleted analytes except cyclohexanone from VOA MDL Table for Project Specific Analytes.</li> <li>17. Added BTEX in Soil to VOA-SIM MDL Table</li> <li>18. Added Manchester MDL to PCB Table</li> <li>19. Updated Skydrol/BHT Control Limits</li> </ol>
11-024	7/19/04	<ol style="list-style-type: none"> <li>1. Revised and Updated MDL Tables for TPH Analyses of Soil/Sediment.</li> <li>2. Revised and Updated MDL Tables for PCB Analyses. Combined All PCB</li> </ol>



		<p>MDL into One Table.</p> <ol style="list-style-type: none"> <li>Deleted all other MDL tables</li> <li>Updated MDL for VOA analysis of Soil using ARI's In-house Method.</li> <li>Added 1-Methylnaphthalene to SIM-PNA MDL Tables for Water &amp; Soil</li> <li>Updated Appendix D (Lab Equipment) and added GeoTech Section</li> <li>Combined Water &amp; Soil SIM-PNA MDL Tables into One Table</li> <li>Deleted Water-SF &amp; Soil SIM-PNA MDL Tables</li> <li>Updated MDLs for Pesticide – Manchester Extraction</li> <li>Revised VOA Water Control Limits Table</li> <li>Updated MDLs for VOA analysis of Water-8260B-5mL purge</li> </ol>
11-023	7/6/04	<ol style="list-style-type: none"> <li>Corrected Conventionals MDL/RL Table</li> <li>Corrected Control Limit for TPH-D MS Recovery in Water Samples.</li> <li>Updated MDLs for NWTPH-D Soil ASE &amp; MicroTip.</li> <li>Removed HPLC MDL Table for analysis of PNA.</li> <li>Removed MDL Table for HCID</li> <li>Removed FID-3B from TPH MDL Tables</li> <li>Updated MDLs &amp; Modified Table for SVOA-PSEP analysis of Sediments</li> <li>Revised Section 11</li> <li>Updated MDL for VOA (524.2) analysis of Water</li> <li>Removed MDLs for VOA-SIM analysis of Soil</li> <li>Updated MDL Table for VOA-Water 20 mL</li> <li>Updated MDL Table for VOA-Water 5 mL</li> </ol>
11-022	5/17/04	<ol style="list-style-type: none"> <li>Corrected Extract Final Volume in MDL table for Sediment PCB</li> <li>Deleted FINN 8 from all MDL Tables</li> <li>Corrected RL for Hg in Water.</li> </ol>
11-021	5/07/04	<ol style="list-style-type: none"> <li>Implemented default control limits for EPA Method 524.2</li> <li>Decreased RL for Aroclor 1221 to level of other Aroclors</li> <li>Eliminated Control Limits for VOA using ARI SOP 804S.</li> <li>Updated VOA 8260B full scan control limits for water &amp; sediment/soil</li> <li>Updated 10 mL purge VOA-SIM control limits for water</li> <li>Changed effective date for VOA-SIM BTEX control limits</li> <li>Updated 8270-SIM-PNA control limits for water &amp; sediment/soil</li> <li>Updated BTS control limits for water &amp; soil.</li> </ol>
11-020	4/26/04	<ol style="list-style-type: none"> <li>Updated MDL (PID1 &amp; 2) for BTEX in water</li> <li>Updated MDL (PID 1) for gasoline in water</li> <li>Deleted MDL Table for ASE extraction of chlorinated pesticides</li> <li>Updated MDL for VOA water 5 mL purge 8260B on NT3</li> <li>Updated MDL for pesticide in water separatory funnel on ECD3</li> <li>Added MDL Table for VPH in water and soil</li> <li>Deleted Control Limit Table for HPLC PNA</li> <li>Updated PCB control limits</li> <li>Updated Herbicide control limits</li> <li>RL for Sulfate to 2.0 &amp; 20.0 ppm for water &amp; solids respectively</li> <li>Updated TPH-D Control Limits</li> <li>Updated Chlorinated Phenols Control Limits</li> <li>Updated BTEX &amp; TPH-G Control Limits</li> <li>Corrected Pesticide MTCA MDL Table</li> <li>Corrected RL for GC-ECD analyses of HCB &amp; HCB</li> </ol>
11-019	3/11/04	<ol style="list-style-type: none"> <li>Revised holding time for Total Solids in soil &amp; sediment from 7 days to 14 days.</li> <li>Updated MDLs for SVOA water L/L NT4 &amp; NT 6.</li> <li>Updated Metals IDLs and MDLs</li> <li>Added QA Policy 9 – Modifications to method, protocol or reports</li> <li>Updated Conventionals MDLs</li> <li>Added QA Policy 10 – Reporting of dual column GC analytes</li> </ol>
11-018	1/21/04	<ol style="list-style-type: none"> <li>Revised Control Limits for GC-MS analysis of SVOA</li> <li>Revised Control Limits for Chlorinated pesticides</li> </ol>



		<ol style="list-style-type: none"><li>3. Updated Appendix E – Table of SOPs</li><li>4. Updated and Revised Appendix F – Sample Containers, Preservation and Holding Times</li><li>5. Modified Sign-of Sheet to include only QA manager</li></ol>
11-017	1/4/04	<ol style="list-style-type: none"><li>1. Minor revisions to Section 13</li><li>2. Revisions to subcontracting language in Section 6.3</li></ol>